REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

AD-A224 644

1. AGENCY USE ONLY *(Leave blank)*	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	May 1990	XXXXXXXDissertation

4. TITLE AND SUBTITLE	5. FUNDING NUMBERS
Application of Computers for Experiment Design, Data Acquisition, and Analysis in the Chemistry Laboratory	

6. AUTHOR(S)

Ronald Paul Furstenau

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)	8. PERFORMING ORGANIZATION REPORT NUMBER
AFIT Student at: Montana State University	AFIT/CI/CIA -90-15D

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)	10. SPONSORING/MONITORING AGENCY REPORT NUMBER
AFIT/CI Wright-Ptatterson AFB OH 45433	

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT	12b. DISTRIBUTION CODE
Approved for Public Release IAW AFR 190-1 Distribution Unlimited ERNEST A. HAYGOOD, 1st Lt, USAF Executive Officer, Civilian Institution Programs	

13. ABSTRACT *(Maximum 200 words)*

DTIC
ELECTE
AUG0 1 1990
S B D

14. SUBJECT TERMS	15. NUMBER OF PAGES
	261
	16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASSIFIED			

APPLICATION OF COMPUTERS FOR EXPERIMENT DESIGN,

DATA ACQUISITION, AND ANALYSIS IN THE

CHEMISTRY LABORATORY

by

Ronald Paul Furstenau

A thesis submitted in partial fulfillment
of the requirements for the degree

of

Doctor of Philosophy

in

Chemistry

MONTANA STATE UNIVERSITY
Bozeman, Montana

May 1990

APPROVAL

of a thesis submitted by

Ronald Paul Furstenau

This thesis has been read by each member of the thesis committee and has been found to be satisfactory regarding content, English usage, format, citations, bibliographic style, and consistency, and is ready for submission to the College of Graduate Studies.

MAy 9, 1990
Date

John K. Craven
Chairperson, Graduate Committee

Approved for the Major Department

May 1, 1990
Date

Edwin H. Abbott
Head, Major Department

Approved for the College of Graduate Studies

May 29, 1990
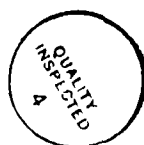Date

Henry L. Parsons
Graduate Dean

STATEMENT OF PERMISSION TO USE

In presenting this thesis in partial fulfillment of the requirements for a doctoral degree at Montana State University, I agree that the Library shall make it available to borrowers under rules of the Library. I further agree that copying of this thesis is allowable only for scholarly purposes, consistent with "fair use" as prescribed in the U.S. Copyright Law. Requests for extensive copying or reproduction of this thesis should be referred to University Microfilms International, 300 North Zeeb Road, Ann Arbor, Michigan 48106, to whom I have granted "the exclusive right to reproduce and distribute copies of the dissertation in and from microfilm and the right to reproduce and distribute by abstract in any format."

Signature _____

Date _____

Accession For	
NTIS GRA&I	☑
DTIC TAB	☐
Unannounced	☐
Justification	
By	
Distribution/	
Availability Codes	
Dist	Avail and/or Special
A-1	

ACKNOWLEDGMENTS

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF FIGURES -- continued

LIST OF FIGURES -- continued

LIST OF FIGURES -- continued

LIST OF FIGURES -- continued

LIST OF FIGURES -- continued

ABSTRACT

The objective of this research project was to develop an introductory chemistry program which uses computers in the laboratory for experiment design, data acquisition, and data analysis. The project used the Montana State University (MSU) laboratory interface system. The system is an integrated hardware and software package which allows for a wide variety of measurements (pH, temperature, time, voltage, current, etc.) totally under the control of the user. The MSU lab interface system was designed to allow students to participate in the process of science. The hardware and software contain components which allow students to design experiments, acquire data in methods which they choose, and organize and analyze the data to draw conclusions.

To meet the objective, we developed and accomplished the following: (1) we developed a model which explains how computers in the laboratory can improve a student's overall understanding of chemistry and laboratories; (2) we developed a laboratory program which successfully taught students how to use the MSU laboratory interface system to design their own experiments; and (3) we developed, tested, and evaluated experiments designed to utilize the capabilities of the MSU lab interface system.

We successfully integrated the MSU laboratory interface system into our general chemistry laboratories. Our students were able to acquire the conceptual knowledge, lab skills, and computer skills necessary to complete an independent design project using the MSU lab interface system. Students overwhelmingly supported the concept of final projects and found the computers useful for learning chemistry in the laboratory. Our approach to using computers in the chemistry laboratory is unique in the country. A comparable integrated hardware/software system and set of curriculum materials simply does not exist elsewhere on any scale, large or small. Computers are no longer viewed as a novelty or as something "neat" to have in the laboratories at MSU; they are an integral part of the chemistry labs.

CHAPTER 1

INTRODUCTION

This thesis represents the compilation of the methods and efforts used to integrate computers into the general chemistry laboratories at Montana State University (MSU). In Chapter 1, I set the stage for the purpose and details of our work by examining the role of computers in the laboratory, detailing some of the history behind the use of computers in chemistry, and discussing some of the basics behind computer interfacing. This chapter lays the foundation for the approach taken in our effort. Chapter 2 highlights the specific objectives of this thesis project. In Chapter 3, I describe a model for using computers in the chemistry laboratory, based on the process of science. This chapter establishes our method and rationale for using computers in the laboratory. Chapter 4 summarizes the hardware and software capabilities of the MSU lab interface system. This system is the focal point for our laboratory development. In Chapter 5, I detail the development of our general chemistry laboratory program, starting with workshops and a pilot program and finally going full scale. Chapter 6 follows with a discussion of several experimental methods which illustrate the capabilities of the MSU laboratory interface system. Finally in Chapter 7, I summarize the results of our computer-based laboratory program, with *the emphasis on student feedback.*

What Chemistry Needs from Computers in the Laboratory

No one can argue the fact that computers are playing an increasingly important role in chemistry and society in general. The impact of computers on today's chemistry laboratories is overwhelming. Almost all modern chemical instrumentation

is now computerized in one way or another. Designers of mass spectrometers, nuclear magnetic resonance spectrometers, and scores of other instruments find computers invaluable for acquiring data quickly and analyzing the data once it's obtained. Computer-controlled signal processing and detectors have dramatically increased instrument precision and sensitivity. Moreover, computers have greatly assisted the users of such instrumentation by increasing the control of experiment variables.

Chemistry certainly benefits greatly by the creativity which computers can help foster in the laboratory. In a manner which will be discussed later in this thesis, computers can assist in the design of experiments. The power of rapid (or slow) data acquisition and versatile data analysis allow the experimenter to do research more quickly and to discover scientific relationships not previously possible without the computer.

It may appear from the first two paragraphs of this section that the computer has become a tool only for the modern chemical researcher . . . *this is not true!* Computers can also significantly improve the manner in which students learn chemistry. Through the interfacing of personal computers with experiments, computers can better involve students in the process of science. Computers can allow students to accomplish laboratory tasks which could not be done otherwise. In short, chemistry needs computers in the student laboratory to provide new opportunities for learning modern experimental methods and experiment design. The majority of this thesis will describe the development of a chemistry laboratory program at Montana State University (MSU) involving the use of computer-experiment interfacing. I will focus on the *development of a model* explaining how computers in the laboratory can improve a student's overall understanding of chemistry and laboratories, the *organization of the laboratory program*, and the *development of experiments* using the MSU laboratory interface system.

Before I begin discussing the details of my research project, I will use the introduction to discuss the historical role of computers in the chemistry laboratory, some basic interfacing concepts and systems, and the role of data analysis in computer interfacing. This background material will lead into the statement of my research problem in Chapter 2.

Computers in Chemistry: A Brief History

Computers have found many roles in the teaching of chemistry over the years. The role of the computer can be split into two general categories: review and investigative. *Review* refers to the use of tutorial and drill software programs for better understanding chemical concepts. *Investigative* refers to using computers to perform calculations, simulate experiments, acquire data, analyze data, and design experiments.

Review

Having computers in the laboratory implies that the computers are available to help not only in the experimental portion of chemistry, but the conceptual part as well. Tutorial and drill software has long been available for a variety of purposes. Castleberry and Lagowski (1) were probably the first to use computers for tutorials on a large scale back in 1970. Tutorial and drill programs are basically used as a method of providing individualized instruction. In chemistry, *tutorial* programs are used as a step-by-step method of teaching about chemical concepts. For example, a gas law tutorial may begin by presenting written information on the screen, and perhaps actively illustrating the basic concepts behind Boyle's and Charles' laws. The user of the tutorial may then literally "page" through the information, much like a book. A computer tutorial has the advantage over a book, however, of being able to "demand" interaction by asking questions. A gas law tutorial may then lead the user into combining the gas laws into the ideal gas law, perhaps eventually requiring the student

to solve gas law problems. The tutorial may now become a *drill* program, posing a series of problems which the student may answer. Most drill programs provide a variety of problems on a subject. Some drill programs explain to the users how they are doing problems incorrectly and what must be done to get on the right track. Since most tutorials contain components of drill programs and visa-versa, I will now refer to tutorial and drill programs collectively as tutorials.

It has only been in the past 10 years, coinciding with the proliferation of personal computers, that the variety and quality of tutorial software has made it a viable option for teaching and learning chemistry in almost any school. In almost every chemistry department in every major college and university, some faculty member has been involved with their own development of tutorial software for their chemistry undergraduates. Since 1981, the Journal of Chemical Education has had no less than 50 articles pertaining to new developments in computer-assisted instruction (CAI) in general, physical, organic, and biochemistry.

The commercialization of many tutorials has done much to make CAI a viable teaching tool. Companies such as HRM Software, COMPress, and Intellectual Software have done a great deal in ensuring that well-tested, debugged tutorials are making their way to general availability. The importance of quality control that is obtained through commercialization cannot be overestimated. Easy to run, "idiot-proof" software is a must for wide-spread use of tutorial software in a chemistry curriculum. No professor would consider using a poorly proofread, error-riddled book in a course; likewise, users have a right to expect the best out of commercially available software. To illustrate the potential usefulness of tutorial software for teaching chemistry, I will discuss a few of the highest quality, most widely used tutorials that are currently available.

KC?Discoverer. Project SERAPHIM, a clearinghouse for information about the use of personal computers in chemical education, has been instrumental in

providing software to interested users at nominal fees (2). Sponsored by the National Science Foundation and the Department of Education, Project SERAPHIM has become a center for the collection, development, and distribution of chemistry software. Perhaps the best product to date to come out of Project SERAPHIM is the multipurpose KC?Discoverer tutorial (3,4). KC?Discoverer allows users to explore the periodic table by graphing and tabulating numerous properties of the elements. Periodic trends in properties such as electronegativity and atomic radius can be seen graphically, quickly and easily. This computerized "CRC Handbook" allows anyone with an interest in chemistry to "dig in" to the periodic table. In fact, we (at MSU) have integrated KC? Discoverer into our introductory chemistry laboratory program to compare with experimental values for electrical conductivity (5).

Because of the clearinghouse function of Project SERAPHIM, it is not possible to thoroughly test and debug all of the software which they distribute. Much of the software has little or no documentation. Project SERAPHIM provides an extremely useful function, but the software distributed is often not usable on a large scale. Commercial software is beginning to provide the quality of programs needed for undergraduate chemistry.

Some Commercial Software. In my opinion, the best tutorial software now available for general chemistry has been developed by Dr Jack Weyh and co-workers at Western Washington University. Included in their bank of software is the "Tools of Chemistry" series, a set of tutorials dealing with such topics as scientific notation and significant figures (6). Much of the reason for Weyh's success lies in the philosophy behind the development of his software. As Dr Weyh puts it, "Drill and practice has a negative reputation. [We must] develop something that the students will enjoy" (7). The big advantage of Weyh's tutorials is that they emulate the paper and pencil logic that students already have. Everything a student needs is at his or her finger tips: a "pop-up" calculator, periodic table, and help screens. The special use of keys is

explained quite well. In addition, the logic and flow is exactly the same in all of Weyh's tutorials, making them all easy to use once the basics are learned.

Another highly regarded chemistry software series is "Introduction to General Chemistry", produced by Dr Stanley G. Smith's group at the University of Illinois (8,9). The series includes more than fifty instructional programs and must be regarded as the most comprehensive series currently available. As with Weyh's software, Smith's series has very consistent operating instructions from program to program. Many of the programs make excellent use of color for illustrating chemical reactions. As with many other tutorials, the Smith tutorials are slow. Slowness is an advantage to a student who needs the extra time in the learning process, but a nuisance to a student who simply needs extra practice.

Tutorials or *review* are but one use of computers in chemistry. In many instances, however, it seems as though a student would be much better off if he or she simply used the time spent on tutorials to work homework problems from their textbook! But these tutorials can provide a tireless, patient mechanism for teaching students fundamental chemical principles. In that regard, the proper integration of tutorials into a chemistry curriculum can help to fill many gaps. But for the chemistry laboratory, the *investigative* role of the computer is a more valuable role than that of review.

Investigative

The *investigative* role of the computer in the laboratory can provide the main assistance in exploring the experimental aspects of chemistry. The first use of computers in the investigative role was for providing computational support for experimental data and theoretical calculations, certainly pre-dating computerized tutorials. As early as 1959, Swinnerton and Miller discussed using digital computers to solve complex chemical equilibrium problems (10). By the mid 1960's, computational methods for acid-base chemistry, equilibrium, and kinetics became more common

(see, for example, references 11, 12, and 13). Physical chemistry was also an early beneficiary of these computational techniques, with molecular orbital calculations becoming a common topic (14,15). In fact, physical chemistry has been one of the most progressive areas of chemistry in terms of using the computer. Entire physical chemistry courses have recently been built around the use of microcomputers to individualize instruction (16). Error analysis and least-squares programs were also early favorites in applying computers to instructional chemistry (17,18).

On the basis of sheer volume of literature, the maturation of computers in chemistry instruction seems to have begun in the early 1970's, coinciding with greater accessibility to mainframe computers and the birth of minicomputers. It should also be noted that during the 1970's the hand-held, programmable pocket calculator also made a significant contribution toward "personalizing" computer use in chemistry. References 19-21 are just a few examples of using programmable calculators in chemistry. It was the 1980's, however, which brought computers into the adulthood of chemistry instruction with the wide-spread introduction of the micro, or personal, computer. Since this thesis involves the use of personal computers in the laboratory, I will focus primarily on post-1980 computer advances.

The introduction of the microcomputer to chemistry has spawned two major development areas within the traditional chemistry laboratory. These areas are *computer simulated experiments* and the *microcomputer-based laboratory*, or *MBL*. (Note: The term microcomputer-based laboratory, MBL, has been borrowed from several sources, including references 22 and 23.)

Computer Simulated Experiments. It can be fairly stated that computer simulated experiments provide the "extreme" alternative to the traditional laboratory. Equipment and chemicals are replaced with the computer and software. Computer simulations create the "no chemicals/no glassware" laboratory. All of the processes involved in laboratory -- the mixing of chemicals, heating and cooling, and control of

apparatus -- are accomplished via the simulation program. Dr. Donald L. Pavia, a leader in the design of computer simulations for chemistry, points out that simulations have two great advantages. First, simulations can focus time, allowing more situations to be covered during a given time period. Secondly, simulations "can relieve students of dependence on their technical and manual skills," which translates into more time to think abstractly about what they are doing (24). As Jones et.al. add, simulations allow students to perform or view reactions "too hazardous, too expensive, or too time-consuming for them to perform in the laboratory" (25). From the literature, 1984 seems to mark the beginning of well-designed simulation programs. The well-designed simulations are highly-interactive, flexible, easy to use, and non-threatening. Computer simulations fall into three categories: *simulated experiments, instrumentation emulators*, and *interactive video.*

With *simulated experiments*, the only hardware required is a personal computer or a terminal tied into a mini- or mainframe computer. Because of the relatively straight-forward calculations involved, acid-base titration curves were among the first simulated experiments in chemistry (26). More recent advances in simulation have emphasized decision making skills. Weyh has developed a simulated inorganic qualitative analysis program which duplicates the thinking skills and powers of observation required in a traditional qualitative analysis laboratory (7). All of the thinking is still left to the students; poor choices can give contradictory results and tests can fail. The simulation does not interpret the results. Similarly, Pavia has developed a simulated organic qualitative analysis program which includes spectroscopic data (24). David Whisnant's simulation, BCTC, provides a scenario in which a hypothetical toxic substance has been released into a river (27). The student must do a simulated analysis of the water to identify the substance and also make economic decisions regarding the control and clean up of the pollutant. These simulations provide color, activity, and help to degrees that will make even the computerphobe smile!

Sophisticated *instrumentation emulators* for personal computers have also been developed. Manual spectrophotometers were perhaps the first instruments to be emulated (29), with development continuing because of these instruments' popularity in general chemistry (30). Not only can instrumentation emulators simulate an actual experiment, but they can also provide valuable, non-threatening experience before ever touching the actual instrumentation. The mainstay tools for organic chemistry -- nuclear magnetic resonance (NMR) spectroscopy, mass spectroscopy, and infrared spectroscopy -- have all been emulated at least to the extent of being valuable training tools for undergraduate chemists. Because of the high costs of such instrumentation, the use of emulators solves time constraint and economic problems, as aircraft operators have realized for decades! Paul Schatz at the University of Wisconsin has developed an outstanding emulator for a Varian EM360 60 MHz NMR (31), while Howard and DuBois have developed a similar emulator for the Varian T-60 NMR (32). Robert Rittenhouse at Eastern Michigan has developed an emulator for High Performance Liquid Chromatography (HPLC) (27). Calculational techniques have also been developed to synthesize mass spectral isotopic abundances, in which simulated spectra are produced (33). In addition, our research group has made initial attempts to use the MSU laboratory interface software to simulate infrared and mass spectra. Instrumentation emulators hold much promise for developing better trained students using modern, complex chemical instrumentation.

Perhaps the most unique approach to computer simulations has been in the area of *interactive video*. Interactive video, primarily with videodiscs, has made it possible to incorporate recorded television images and audio into a computer program. This allows the programmer to combine and overlay computer texts and graphics on the same screen. This technique requires specific and expensive computer and videodisc hardware, described in reference 25. The unquestioned leaders in this technology as applied to chemistry is the group headed by Smith and Jones at the University of Illinois (25). Their group has developed numerous chemical experiment simulations

using interactive videodiscs, including the topics of the physical properties of elements, chemical reactions, and chemical kinetics (34). The interactive videodisc has the advantage of allowing the student to see videos of actual chemical reactions taking place, not computer-generated images which lack realism. This allows students to see chemical reactions not normally performed due to cost or safety issues. In many interactive scenarios, the student can choose what reactants and how much of each reactant to use. The University of California at Los Angeles and the University of Nebraska have also been extremely active in developing videodisc technology for chemistry, developing programs for acid-base titrations and classroom demonstrations (35,36). It is clear that the interactive videodisc is rapidly becoming a usable tool for the chemistry laboratory.

As innovative and useful as computer simulated experiments are becoming, they are still not a direct substitute for laboratory work (25). As Pavia notes, "it is still necessary for the chemistry student to master certain manual laboratory techniques", with the computer simulation enhancing the learning experience (24). MacKenzie points out that simulations "suffer by their abstract presentation of real world phenomena" (22). Students can simply develop a false sense of reality from the simulation of complex phenomena where the possibility of error becomes minimized or non-existent. Computer simulations, then, do not provide the ultimate solution to the implementation of computers into the chemistry laboratory. Treading the water between the traditional laboratory and the simulated laboratory is the microcomputer-based laboratory, or MBL.

The Microcomputer-Based Laboratory. The MBL can be thought of as an intermediate between the simulation at one extreme (abstract, little human involvement), and the traditional experiment at the other (inaccurate, of limited scope, labor intensive) (22). The MBL, also known as laboratory interfacing, uses a computer connected to an experiment to detect and control what is occurring in that experiment.

(I will use MBL and computer interfacing interchangeably throughout this thesis.) A very simplified block diagram of the MBL concept is shown in Figure 1.

| STUDENT | COMPUTER | INTERFACE | EXPERIMENT |
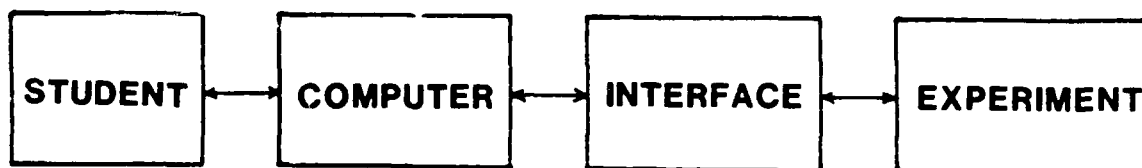
FIGURE 1. The Microcomputer-based Laboratory (MBL): Laboratory Interfacing.

In laboratory interfacing, real world experimental variables are measured or controlled with a variety of sensors. The signals from these sensors (of light, temperature, etc.) are monitored by the interface electronics. The computer provides the data acquisition and control. Unlike simulations, laboratory interfacing is a part of the laboratory environment, not an extension of that environment. In the MBL, computers are *in* the laboratory, not outside of it.

Of all of the uses for computers in the chemistry curriculum, laboratory interfacing is unquestionably the least developed. Although computer manufacturing variety (non-standardization) and other hardware factors have contributed to underdevelopment, the most probable reason is economic. In the past, the cost of competent microcomputers and complexity of interfacing made it economically impractical on a large scale. Within the last 5 years, however, inexpensive and powerful microcomputers have made the MBL an economically attractive alternative in laboratory modernization. As will be developed in this thesis, the MBL is attractive from a pedagogical standpoint as well.

The MBL has many advantages over the traditional laboratory and computer simulations. First of all, *computers in the laboratory expose students to the way science is really done in the 1990's!* Nearly every research effort in chemistry and physics

involves the use of computers for data acquisition, analysis, instrument control, or information retrieval. It is the disadvantaged student indeed who happens to drudge through graduate school in the sciences today without computer experience! Why shouldn't we use computers for the same functions in our undergraduate laboratories?

Secondly, *computers can gather large amounts of data rapidly and accurately.* This advantage creates the opportunity to design experiments and study chemical concepts which would normally not be possible in the traditional laboratory. More complicated and challenging experiments can be developed. The computer allows the student to manipulate these massive amounts of data in meaningful ways.

The third main advantage of the MBL provides much of the impetus for this thesis work. The MBL can *help teach students to think independently in the laboratory and to participate in the process of science.* As Marcus Mendenhall puts it, one of the most important skills to learn in the laboratory is "to think about the limits to an experiment or, more generally, the limits to anything with which one might interact in the real world" (37). Simply put, *the MBL allows the student greater control of a laboratory experiment.* As this thesis will develop, the MBL can foster creativity in the laboratory for even the non-scientist. The computer can remove barriers to experimentation while teaching new skills, thus promoting creativity. With time, the computer can become as natural and as important a tool in the laboratory as beakers and chemicals. In addition, the MBL makes it easy to incorporate nearly all of the aspects of computerized chemistry instruction previously described.

The remainder of this thesis will detail the application of computer interfacing in introductory chemistry laboratories. This INTRODUCTION chapter will conclude with sections describing the basics of a MBL, including basic interfacing concepts and data analysis. The remaining chapters of this thesis will detail the application of the MSU lab interface system at Montana State University.

Basic Interfacing

The purpose of this section of the INTRODUCTION is to provide the reader with some background information about different types of computer interfacing. This section will contain very little detailed electronics specifications, though many of the references cited contain this type of information. The text will focus on describing basic interfacing concepts and some of the approaches to computer interfacing found in the literature. The details of the interface used in this research, the Montana State University (MSU) laboratory interface system, will be discussed in Chapter 4.

What is meant by the term *computer interfacing*? In a general sense, computer interfacing means the integration of the computer into the laboratory as a scientific instrument; the microcomputer-based laboratory (MBL). In a specific sense, computer interfacing means connecting a computer to laboratory instruments for data acquisition and control. Certainly, data analysis should be considered a part of computer interfacing. But because of its strictly software nature, I will discuss data analysis separately in the last section of the INTRODUCTION.

The fundamental goal of basic computer interfacing is quite simple: to develop hardware devices which act as sensors in the experimental environment and software which can access these sensors (38). Sensors include devices to measure time, temperature, light, current, voltage, humidity, pH, radiation, or whatever is needed in the experiment. Dr Michael H. Powers, one of the nation's leaders in developing and promoting do-it-yourself interfacing, divides computer interfacing into two categories: connection via the *computer gameport* and via *external circuitry* (38).

Computer Gameport

Historically, the early proliferation of Apple, Commodore, and Atari computers made gameports a popular hardware target for interfacing. This is due to the easy access to input and output signals (39). Gameports are normally attached to a joystick or game paddle for use with video games. A joystick is simply a set of four switches, whereas a game paddle is a potentiometer connected as a variable resistor. Depending on the model of computer, the gameport is either an internal 16-pin input/output (I/O) connector socket inside the computer, or an external 9-pin "D" connector. Each pin has a particular assignment (data flow, ground, +5 volts, etc.) and are discussed in detail in references 40 and 41.

DeJong and Layman have led the way in the development of do-it-yourself gameport interfacing for science teaching (primarily in physics), beginning in the early 1980's (41-43). Their applications, primarily with Apple II computers, include using thermistors, phototransistors, and timers. It should be noted that DeJong and Layman's do-it-yourself approach requires significant skill in electronics and programming in either assembly language or BASIC. However, much of the driving software is readily available and inexpensive. Many others have developed data collection devices and experiments for Apple II computer gameports with a variety of transducers and instruments, with temperature being the most commonly measured quantity (44-53). Similar gameport interfacing projects have been developed for the Commodore VIC-20 and C-64, which include experiments with thermistors, cadmium sulfide light cells, radiation detectors, and colorimeters (54-56). Again, these efforts require considerable programming and electronics skills. Several gameport enthusiasts cite the removal of tedious data collection as a primary reason for developing computer interfaced experiments. In other words, an important reason for interfacing is to save time, allowing students to focus on the analysis and interpretation of their results.

The main advantage of these homemade gameport interface experiments is that they are usually very inexpensive, with basic tranducers and connectors costing less than $10. The main disadvantages, already pointed out, are that considerable programming and electronics skills are often required. In addition, gameport devices tend to be slow. These disadvantages severely limit the flexibility of gameport interfacing. Greater flexibility is offered through *external circuitry*.

External Circuitry

Using external circuitry involves using electronics packages outside of the computer. An external circuit, then, is an *interface* between the computer and the sensing device. The interface conditions signals, translates signals, and essentially allows for communication between the computer and sensing devices. Of course, no communication is actually possible unless there is software which can translate between the interface and the computer. *External circuitry devices will normally allow faster sampling rates and higher resolution than gameport devices.* In addition, external circuitry devices will generally allow multiple sensors, a variety of different sensors, and protection of the internal computer circuitry (38). These devices range from the very simple to the very complex.

A very simple device can be an integrated circuit, along with a few electrical components, wired to a breadboard. More complicated interfaces are prepackaged (like the MSU lab interface) and involve many more components. It should be pointed out that most modern chemical instrumentation, like a nuclear magnetic resonance spectrometer, incorporates very specific interface devices (complete with software) which allow the operator to completely control the instrument.

The fact that Apple and IBM-type microcomputers are bus-based make them ideal candidates for interfacing by external circuitry. A bus is simply a bundle of wires which can pass commands from one part of the computer system to another.

These computers contain a motherboard which houses the computer bus. The system can be expanded for computer interfacing by plugging in boards into the bus (57). The bus-based system makes communication with the computer a reasonably straightforward, though not simple, task.

External circuitry devices can communicate with the computer in a number of ways. These include serial communications (also known as the RS-232 standard link) and parallel communications. The GPIB (general-purpose interface bus), often referred to as the IEEE 488 parallel transmission system, is a popular option for instrumentation control. Annino and Driver provide an excellent discussion of standard computer communication interfaces in Reference 57. Communications can be accomplished via ports already built into the computer, or via specially designed cards which are installed into the computer.

There are many instances in the literature of application- or instrument-specific interfaces designed and built by researchers. References 39 and 58-60 provide a few examples. Some are simple, some are very complex. Commercial vendors, such as Vernier Software (38), HRM Software (38), Champ (61), IBM (23), and Tel-Atomic (62,63) provide completely assembled interface packages over a range of prices. All vendors seem to take a different approach to hardware design and communication methods. Some require the user to program the interface unit using BASIC. Others have prepackaged software designed to take specific measurements, offering little flexibility to the user. From a general sense, key design considerations for data acquisition external circuitry include signal conditioning, transmission cabling, filtering, sampling rates, digital input/output (I/O), and analog I/O (57).

One of the promising aspects of computer interfacing may also be the Achilles' Heel for widespread application: there are *so many ways* to do computer interfacing! A laboratory interface may be very simple, requiring only a few electronic components. Or, the interface may be very complex, requiring intricately designed circuit boards. It is the variety of interfaces, the complexity of the electronics, and

the lack of standardization which has hampered the widespread application of computer interfacing into instructional laboratories (22). Equally important is the lack of versatile, easy-to-use software. It is important to note that we view the MSU laboratory interface as an effort to establish an integrated approach to laboratory interfacing. *This integrated approach requires not only hardware development, but integrated software, experiment, and curriculum development as well.*

Data Analysis

The ability to use a computer to perform rapid data analysis must be viewed as an integral part of the microcomputer-based laboratory. The hardware and software to accomplish data acquisition is the high visibility aspect of the MBL. But it is the software that can analyze, graph, and display the data which is often the most important to the user. Being able to organize data and draw conclusions from that data is an important part of the process of doing science. A data analysis package can make it much easier to see what experimental data *really* means, removing boring and tedious activities such as repetative calculations.

It should not be surprising that there are many examples in the literature of using commercial spreadsheets for data analysis. Commercial, multi-purpose spreadsheets such as Lotus 1-2-3 and Quattro are highly developed, well polished products. These spreadsheets offer a wide range of calculational and graphing capabilities. Levkov gives a good discussion of the uses and capabilities of Lotus 1-2-3 for the science lab (64). Joshsi offers a similar, more detailed discussion of Lotus 1-2-3 specifically for physical chemistry (65). Kari and Dubreuil have used SUPERCALC 3A for an Apple IIe computer in their chemistry laboratories (66). Powers details the use of the Vernier Software voltage input unit and pH electrode to obtain pH titration data for sodium carbonate and HCl (67). The data file is then analyzed and/or graphed using the Vernier Software Voltage Plotter III, AppleWorks,

PROSTAT, and Sigma-Plot spreadsheet packages. IBM has attempted to integrate a data analysis package into their Personal Science Laboratory MBL concept, with mixed results (68).

For the most part, commercial spreadsheets are *not* easy to learn how to use. The quality which makes these spreadsheets so popular is also their biggest drawback for widespread laboratory use. This quality is versatility. Versatility implies many options and capabilities. This can lead to confusion for the user who simply wants to do data analysis and graphing of his or her experimental data. We felt it was necessary, then, to develop a data analysis capability tailor-made to the capabilities of the MSU lab interface hardware and to the abilities of our students. An integrated, easy-to-use spreadsheet should be an important part of any MBL effort.

Although many bits and pieces exist, an integrated approach to using computers in the chemistry laboratory simply does not exist outside of MSU. It is the purpose of this research project to establish a laboratory program and develop experiments around the laboratory interface concepts designed and built at MSU. I will discuss the objectives of my research project (or, a statement of the problem to be solved) in the next chapter.

CHAPTER 2

STATEMENT OF THE PROBLEM TO BE SOLVED

The Objective of the Research Project

As discussed in Chapter 1, there have been many, varied efforts to employ the personal computer into instructional chemistry. However, it is apparent from the literature that the personal computer has not yet been well integrated into the instructional laboratory, especially at the introductory level. Certainly, no researcher has come close to applying computer interfacing to the general chemistry laboratory in a broad, effective manner. In some areas, it almost seems that the computer has been a solution waiting for a problem. In some instances, the computer has been used without really thinking about what it is that needs to be accomplished. The previous statement is in no way meant to diminish the many fine efforts already made in developing the computer as an instructional tool in chemistry. On the contrary, it is meant to underscore the need for careful development of computer usage in instructional chemistry, especially as a laboratory tool. One of the greatest potential uses for the computer as an instructional tool lies in the introductory laboratory. *The objective of this research project is to develop an introductory chemistry program which uses computers in the laboratory for experiment design, data acquisition, and data analysis.*

The Goals of the Research Project

The MSU laboratory interface system gives us the chance to make this opportunity a reality. As will be discussed in subsequent chapters of this thesis, computers offer us a chance to teach laboratory science in the manner in which it is

actually done. This requires developing new curriculum materials which take advantage of computers in the labs. It also requires the "rethinking" of the types of labs we do, taking advantage of the new areas of experimentation which computer-laboratory interfacing-- the microcomputer-based laboratory-- can open up to us. The following goals were developed to meet the objective previously described:

(1) Develop a model which explains how computers in the laboratory can improve a student's overall understanding of chemistry and laboratories.

(2) Develop a laboratory program which successfully teaches students how to use the MSU laboratory interface system to design their own experiments.

(3) Develop, test, and evaluate experiments designed to utilize the capabilities of the MSU lab interface system.

The organization of the remainder of this thesis will follow the presentation of these goals. In Chapter 3, I will discuss a *model* for using computers in the chemistry laboratory. Chapter 4 will follow with a description of the *MSU lab interface hardware and software* and how it compliments the model described in Chapter 3. The *laboratory program* will be discussed in Chapter 5, with a description of *experiments* following in Chapter 6. I will *summarize the results* of this thesis work in Chapter 7.

CHAPTER 3

A MODEL FOR USING COMPUTERS IN THE LABORATORY

One learns science by doing science. A simple statement, but it provides the underlying reason for teaching chemistry laboratories in the first place. It will also provide the rationale for using computers in the laboratory. From this "doing" perspective, science is very similar to art and sports. One becomes good at science by doing science, not just by being a passive observer. Chemistry is very much a "doing" science, a laboratory science.

But how does one *do* science? Most scientists would agree that one does science using the *scientific method* or the *process of science*. Others consider the scientific method as the ground rules within which the game of science is played, not a prescription for carrying out scientific research (69). Nonetheless, the *process of science* provides a framework for a systematic approach to scientific research (70). *The process of science will provide the basis of our model for using computers in the chemistry laboratory.*

The Model: Duplicating the Process of Science

Our main goal in involving computers in our general and introductory chemistry labs is to teach science the way science is actually done. In the process of science, also known as the scientific method, the following iterative process is prescribed (71,72):

(1) Look for unexplained behavior in nature (Identify Problem).

(2) Design experiments which will yield information concerning this behavior (Design Experiment).

(3) Collect this information (Collect Data).

(4) Organize this information to look for cause and effect relationships (Organize Data, Draw Conclusions).

A graphical representation of this process is shown below in Figure 2. The circle and quadrants were chosen to represent the process because the scientific method is a dynamic, interactive process. In reality, science is seldom done in a rigid, step-by-step process. Indeed, it is far more common that one group of scientists propose an important problem to study, with others designing and performing the experiments. Still others may propose theories to account for the results, while other groups design experiments to distinguish between competing theories (69).

PROBLEM —> EXPERIMENT —> DATA —> GENERALIZATION
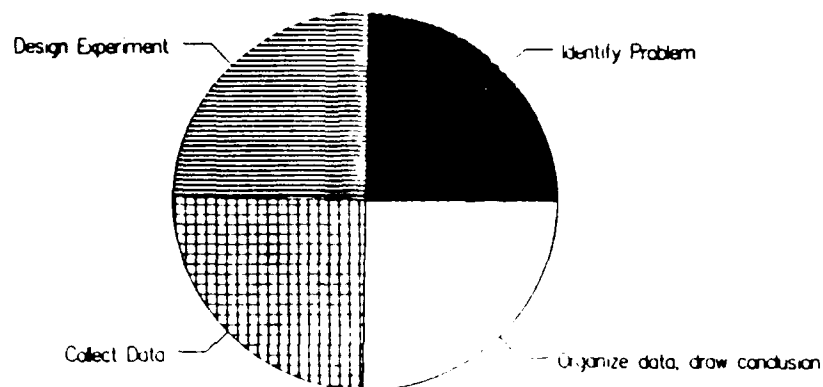
FIGURE 2. The Process of Science.

The process of science is a creative process. As J.V. Mallow states, "Science is not simply a series of cookbook recipes. In fact, as any creative chef knows, neither is cooking simply a series of recipes" (69)! It takes creativity to know what to look for in nature. It takes creativity to design experiments which yield information about behavior in nature. It certainly takes creativity to develop meaning and theories from experimental data. The process of science does not provide the answers; it provides a method for *finding* the answers.

Learning science involves the learning of basic skills. As Chang notes, the study of chemistry involves three basic steps: observation, representation, and interpretation (70). *Observation* skills are learned in the laboratory by seeing chemical reactions and observing the results of chemical and physical phenomena. Important clues to a chemist will likely mean nothing to the average student unless observation skills are developed. A chemist *represents* what he or she observes through the use of chemical equations, mathematical formulas, graphs, and tables. These methods of representing observations communicates the information to others. Therefore to learn chemistry, a student must also learn the methods by which chemists communicate. Finally, chemists *interpret* chemical concepts by proposing theories involving atoms and molecules which they cannot see, but account for their observations. As one can see, using the scientific method involves not only understanding the process, but having the skills to apply it. Figure 3 is a different representation of the process of science shown in Figure 2, emphasizing some of the skills involved in the process. Again, the process of science is an iterative and dynamic one. The steps in solving laboratory problems shown in Figure 3 hold true regardless of whether it is a research lab or a student lab. The only difference is the degree of difficulty and the point at which the student may enter the picture.
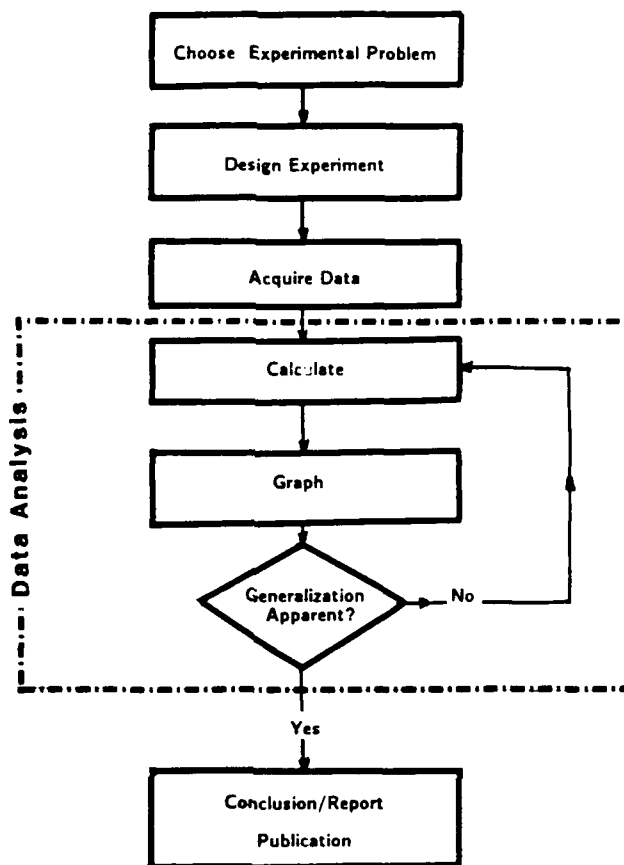
FIGURE 3. The Process of Science in the Laboratory (73).

The Process of Science in the
Chemistry Laboratory

In a research lab, the chemist chooses an experimental problem based on his or her interest and area of expertise. It is very difficult for a student at the introductory level to *identify the problem* which should be the target of his or her laboratory work. In a student lab, the experimental problem has already been chosen based on the concept the instructor wants the students to learn about on that particular day. Lack of time and experience are probably the primary reasons for identifying the problem for the students.

Designing the experiment is probably the most important part of any good research project and perhaps the step which requires the most skill. In addition, it is one of the most exciting parts of doing research. For most students, certainly those at introductory levels in college, the design of the experiment has been completed by the teacher or text author. The design of the experiment appears as a set of directions in a laboratory manual. The manual tells the students the exact equipment and materials to select and sets forth a step-by-step procedure for gathering the information required to reach the desired conclusion. While this "cookbook" approach to laboratory experimentation may effectively reinforce chemical concepts, much of the process of science is missing in it. Why are labs done this way? It is because most lab courses have too little time available, making it necessary to have the student enter at the *collect data* step of the process of science. I'll come back to this point later; this is an area where computers can make a big difference in the laboratory.

Following the directions set out in a lab manual, the students *collect data* and subsequently analyze the data. Data analysis can take many forms: calculations, graphs, or generalizations from simple observations. The data analysis and/or collection continues until the student (or researcher) can *draw some conclusion* about the data. In research, this may include development of a theory which explains the data. Students are often asked to write a report evaluating their results. Researchers will publish their results in a journal.

Integrating Computers into the Process of Science

Our model for using computers in the laboratory is essentially that of the process of science. Computers have a place in the laboratory when they can do one or both of the following:

(1) Involve the student in *more* of the scientific process (see Figures 2 and 3).

(2) Allow the student to do any step in the process of science *better, faster,* or *more accurately.*

Computers should not be thought of as gadgets or something "neat to have" in a laboratory. If the computer application in the laboratory offers no enhancement over a conventional laboratory, don't use it! Students must see the computer as an everyday tool in the lab, allowing them to accomplish tasks that are difficult, if not impossible, without the computer.

Computers can help bring even introductory students into more steps of the process of science. Given a set of experimental objectives, a computer in the lab can be the tool that involves the student with the design of the experiment, the gathering of information, and the organization and analysis of this information. A well-integrated lab interface software/hardware package can individualize how a student approaches all aspects of an experiment.

Since the focus of a laboratory is on experimentation, students should have many options with respect to the type of measurements which can be made, the time scale on which the measurements are made, how the measurements are displayed or stored, and how the information can be organized and analyzed once obtained. Computers can rapidly and accurately acquire a wide range of data from an experiment and package it in a variety of ways. A computer/lab interface (MBL) can replace a wide variety of instrument systems. A personal computer can provide a power supply, information storage and processing, and a readout mechanism. A laboratory interface unit can provide signal conditioning amplifiers and timing and counting circuits for a variety of measurement-specific detectors (temperature probes, pH probes, currents, voltages, Geiger counters, etc.). The computer can truly become a student's "lab partner", allowing the student to participate in all aspects of the process of science.

To be effective, the computer must be an integrated part of the instructional laboratory. As shown in Figure 4, a good laboratory interface system is the key to successfully employing computers in the laboratory. A laboratory interface, with many adaptable detectors, can allow the student to take many combinations of data at

rates not previously possible in a normal chemistry lab. A good system must give the student control, via the keyboard or switches, of the experiment itself and how the data is acquired. In addition, since the computer is already in the laboratory, software for analyzing and graphing the data should also be available. Since tutorial and drill software are available, the lab computer also allows students to practice their problem solving skills. Also, the computer can be used to simulate experiments which are too dangerous or too expensive to perform. Computers in the chemistry laboratory create an environment of individual experimentation and flexibility previously unavailable.
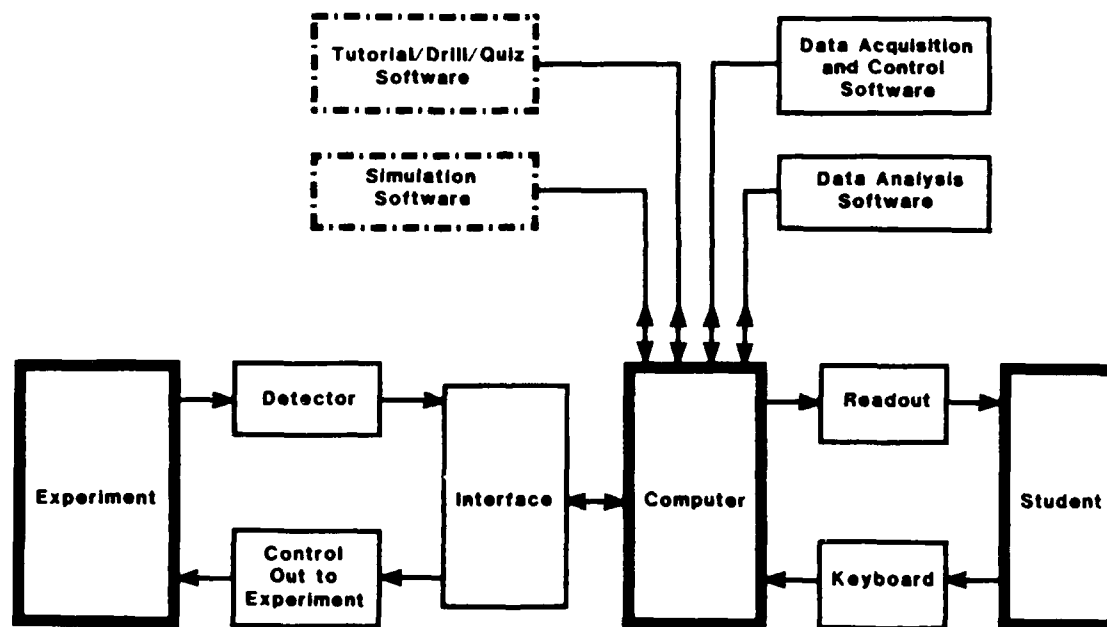
FIGURE 4. The Personal Computer in the Laboratory.
Software support is the key to multiple uses of the
computer in the laboratory.

We have designed the MSU laboratory interface system hardware and software to involve students in the process of science, as shown in Figures 2 and 3. The specifics about how the system does this will be discussed in Chapter 4. One of the

main reasons the MSU interface system allows for student participation in the process of science is that it *redistributes the time in the laboratory.*

Redistribution of Time

To illustrate how computers can reallocate the time spent in a laboratory, I will refer back to the pie chart presented in Figure 2. Figure 2 simply illustrates the process of science in four equal quadrants. This pie chart also is a good vehicle for displaying the amount of time spent in the lab for each of the four areas. Figure 5 represents, based on our experience, our perception of how students' time is spent in a conventional, non-computer based laboratory. As one can see, the majority of the time is spent collecting data; a task that, though important, is not very thought-intensive. Since most laboratory books give carefully outlined procedures, little (if any) time is spent actually designing the experiment. The large amount of time spent gathering data leaves much less than adequate time available to organize the data and to draw meaningful conclusions.
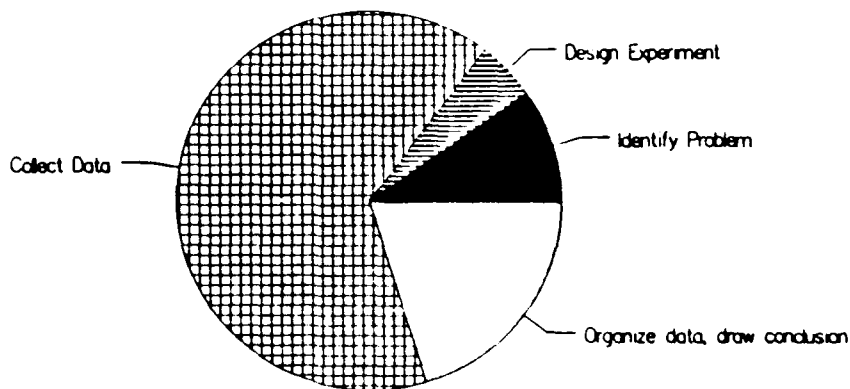
FIGURE 5. Time Allocation in a Traditional Chemistry Lab.

We have succeeded in redistributing the time that a student spends in the laboratory from tedious activities to thought-provoking activities. We have developed a laboratory program, described in Chapter 5, in which students are allowed to explore a variety of chemical concepts by designing their own experiment, using the computer interface system, to meet the objectives. The tedium is taken out; the thinking is put in! As I. Scott MacKenzie notes,

> The 18th century German philosopher and physicist Liebniz said, "It is unworthy of excellent men to lose hours like slaves in the labor of calculation." Unfortunately these wise words are not heeded in many cooked experiments; not only are students engaged in non-exploratory pre-designed activities, they are often required to gather legions of data and perform extensive and tedious calculations on them. Such drudgery is unlikely to inspire students to pursue careers in science. Thus another need exists for students to get on with the analysis and interpretation of results-- more thought-provoking activities-- and bypass, or at least expedite, the gathering of data (22).

By incorporating the MSU lab interface system into our laboratories in conjunction with research projects (described in Chapter 5), even the introductory student can become more deeply involved with the portions of the process of science that require the most thought. As shown in Figure 6, the integrated hardware/software system of our computer based laboratory has allowed us to emphasize the areas of experiment design and the organization of data, the areas of the process of science which require the most thought. Much less time is now spent on the collection of data, which the computer interface system can do much more easily, accurately, and effectively than in a traditional laboratory.

The combination of the lab interface and the research/design project allows us to realize the time allocation shown in Figure 6. This time distribution is based on our perceptions of student activity in the laboratory. The students are more involved in the process of science because they are now designing experiments and have more time available for the thought-intensive aspects of experimentation.
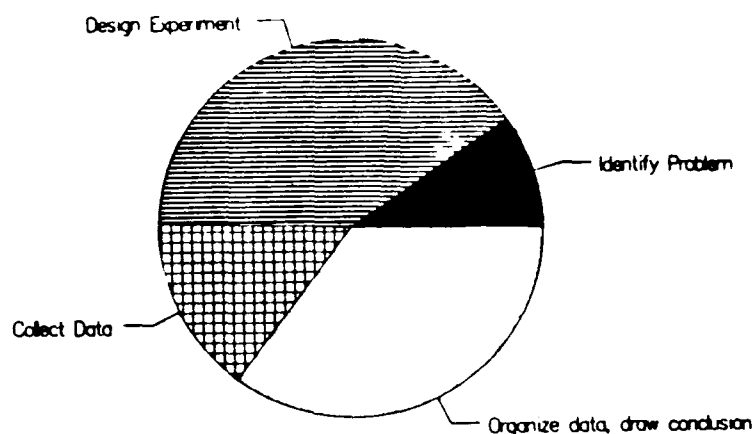
FIGURE 6. Time Allocation in the MSU Computer-based Chemistry Lab.

Necessary Components of the Model

Our model for using computers in the general chemistry laboratory is to assist students in following the process of science. As I will show in Chapter 4, the MSU lab interface hardware and software are designed to duplicate the steps involved in the process of science. We have designed a laboratory program (Chapter 5) which takes advantage of the computer/interface capabilities to shift more time into the thought-intensive aspects of the scientific method. It is important at this time to briefly discuss the skill components which must be developed to effectively deploy the model. These skill components, which will be discussed in more detail in Chapter 5, are *conceptual skills, laboratory skills,* and *computer skills.* All of these skills are essential toward making the process of science in the computer-based chemistry lab work.

Conceptual skills refer to developing those skills necessary to understand basic chemical concepts. The lecture component of a chemistry course goes a long way

toward developing those skills. It is often useful for a laboratory and lecture course to be complementary and timely. Concepts learned in lecture can then be immediately applied to the laboratory and visa versa. Laboratory time can (and should!) make concepts discussed in lecture come alive. Knowledge of basic chemical concepts provide a foundation upon which true discovery can actually take place.

Laboratory skills includes proper use of equipment and glassware, the making of solutions, safety, and so on. This also includes developing observation and technique skills. Learning proper experimental technique and attention to detail is extremely important. Students must learn what to look for in an experiment, how to control variables, and how to take good notes during an experiment. Most students may perceive that they are in lab only to experience performing laboratory procedures. However, lab skills are only a portion of the skills needed to realize the process of science.

In the computer-based laboratory, developing *computer skills* becomes very important. Many critical questions now begin to surface which have never before needed to be addressed in the laboratory. It is obvious that *some* basic computer skills are needed when computers are employed for any task, but to what extent? How much should students know about DOS? Should they be *required to do programming*? How much time does learning how to use the computer consume? Are we teaching more computer science than chemistry? Are computers really useful for learning chemistry in the laboratory? These questions must be addressed when developing a microcomputer-based laboratory.

The purpose of using computers in the laboratory *must* be to better involve the students in the process of science. Unlike many other academic disciplines, chemistry requires the learner to *do* chemistry, rather than just read about it. The computer can change the way we do chemistry, for the better, in the instructional laboratory.

CHAPTER 4

THE MSU LABORATORY INTERFACE HARDWARE AND SOFTWARE

The purpose of this chapter is to describe the MSU laboratory interface hardware and software and to show how the design incorporates the model described in Chapter 3. Much of the work described in this chapter has already been discussed by members of this research group in several journal articles (71-74). This chapter will present much of the same information, including more details.

The MSU laboratory interface project was conceived in 1986. The initial motivation for the project was economic. Funding had become available to update the instructional laboratories. However, with 60 laboratory stations, the cost of purchasing quality pH meters, colorimeters, Geiger counters, precision thermometers, and other equipment for each station was well beyond the budget. At this time, high performance personal computers were available for about $1000. Because of the low cost of computers, this research group (headed by Dr John Amend) decided to design and build a computer-based laboratory interface system which could make all of the instrumental measurements required in a chemistry course. In addition, the system would provide data logging and data analysis. The Department of Chemistry acquired 45 Zenith Z-158 computers using the newly available MSU student computer fee funding, while Zenith Data Systems donated 15 additional Z-158's. University funds were used to construct the 60 lab interface units. The total cost of each laboratory station, with interface and computer, was less than $1500. This chapter will detail the design of the laboratory interface *hardware* and *software*.

Hardware

The Interface as an Instrument

The MSU laboratory interface, from a hardware standpoint, must be thought

of as a multi-purpose laboratory instrument. As discussed in references 73 and 75, the

role of a laboratory instrument is quite simple: to interact with a physical or chemical

quantity of interest, and to transfer information concerning this quantity to a student

or researcher. All laboratory instruments are designed in basically the same way to

fulfill this role. As shown in Figure 7, a measurement-specific detector interacts with

the quantity of interest. The detector produces a small electrical signal related to the

magnitude of the quantity being measured. The signal is then conditioned by

amplifier circuits and passed on to a data processor. The processor performs algebraic

operations on the signal, ultimately resulting in a number displayed to the observer on

a readout device.
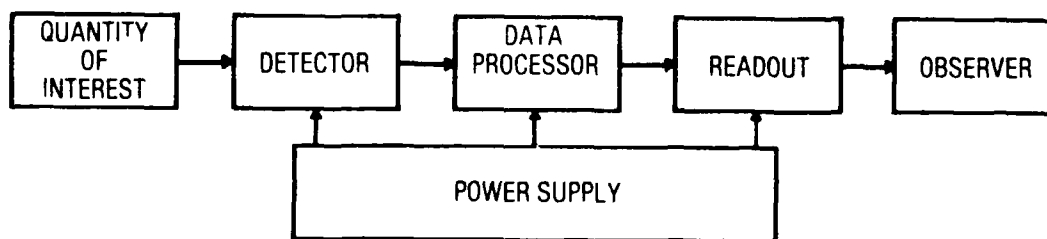
FIGURE 7. Components of a Laboratory Instrument.

In the generalized laboratory instrument shown in Figure 7, the detector (with

signal conditioning amplifier) and the mathematical algorithm performed by the data

processor are measurement specific. The data processor itself, the readout, and the

power supply are generic components of all instruments (75). Although specific

instruments (such as a pH meter) use specialized circuits to perform algebraic

operations on signals, a personal computer can perform this function with the

appropriate software. This is illustrated in the inner dashed box in Figure 8. The function of the lab interface, then, is to provide the circuitry to acquire and condition the detector signal.
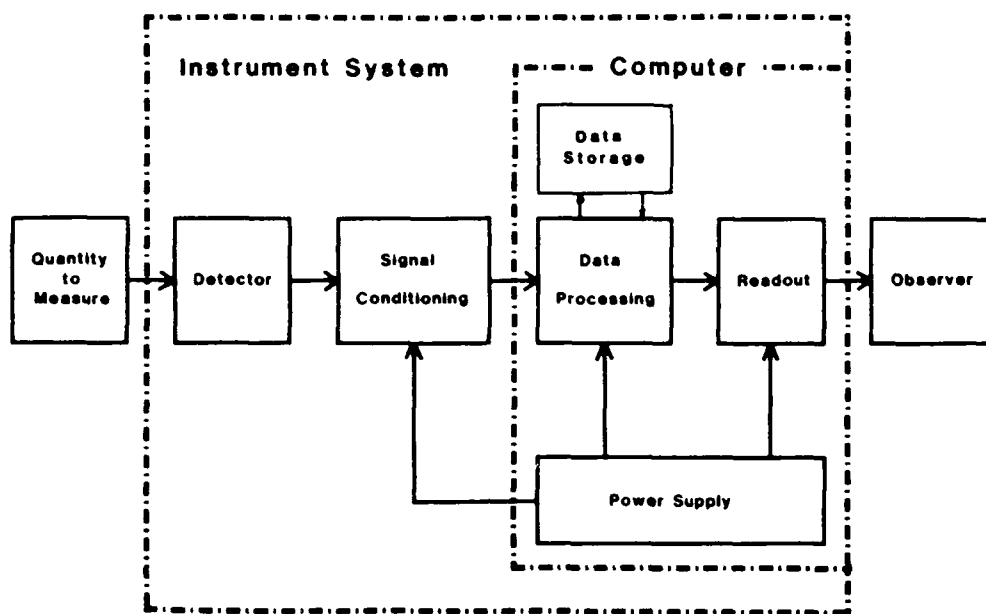
FIGURE 8. The Computer as Part of an Instrument System (75).

The MSU Lab Interface

In undergraduate chemistry laboratories, a relatively small number of quantities need to be measured precisely. These include temperature, light, voltages, currents, counts, pH, and time. The MSU laboratory interface system replaces the signal processing units and power supplies of the instruments required to make these measurements, adapting to many different detectors.

Several criteria were involved in the design of the MSU interface and its support software:

(1) The system should support the process of science in experimentation.

(2) To be economic, it should make *all* of the electronic measurements required in lower division college science laboratories.

(3) It should make these measurements with research precision.

(4) It must be easily programmed, so that students (and instructors) can concentrate on science rather than computer programming.

(5) It must be able to participate in simulated experiments, giving students control of the system and provide time-based information for real-time simulation experiments.

(6) It must be expandable at low cost to provide for additional functions not yet envisioned.

A photo of the MSU lab interface is shown in Figure 9, with a temperature sensor attached. A smaller version of this interface was developed in 1988 with half the inputs and outputs. A redesigned interface was developed in 1990 which is essentially a hybrid of the large and small versions, with increased resolution.
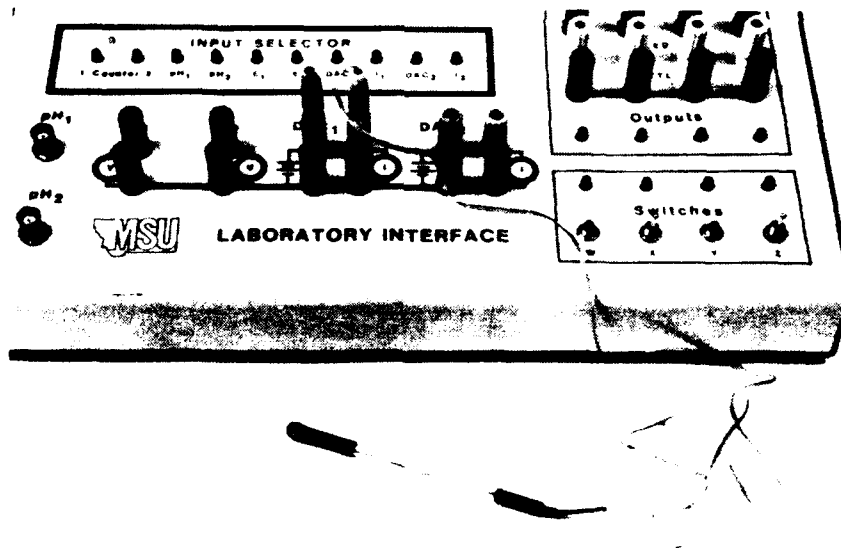
FIGURE 9. The MSU Laboratory Interface.

To communicate rapidly and accurately between the computer and the interface box, a card carrying a parallel port was developed. Cards were developed for both IBM-compatible and Apple II computers. A ribbon cable runs from the card to a nine-pin "D" connector on the back of the interface. This connection provides power (+5 and +/- 12 Volts) and three programmable logic lines for control of external multiplexers, instrumentation amplifiers, and accessories yet to be designed (73).

Figure 10 is a block diagram of the MSU lab interface electronics. The interface unit used in the MSU laboratories is capable of receiving input from two pH probes, two voltage inputs, two current inputs, two Geiger counters, and four digital photodetectors. It can also send digital signals to two digital-to-analog converter (DAC) outputs and digital signals to four transistor-transistor logic (TTL)/light-emitting diode (LED) outputs and an auxiliary port (76).
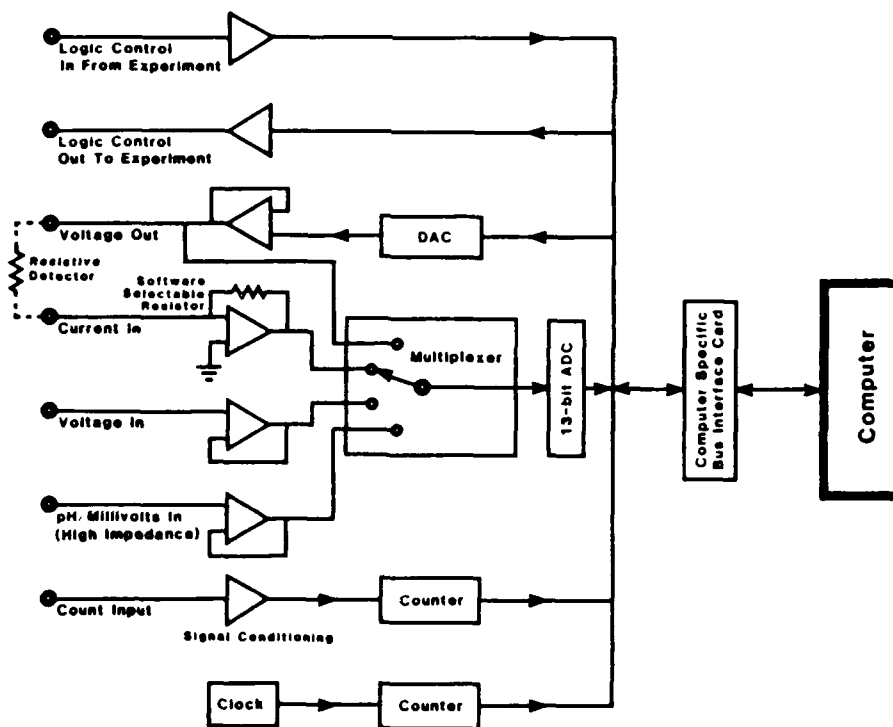
FIGURE 10. Block Diagram of the MSU Lab Interface Electronics (73).

As seen in Figure 10, the interface contains several operational amplifier circuits. Voltage followers provide high impedance inputs for pH electrodes and voltage measurements. The pH can be measured between 2 and 12 pH units, with resolution of 0.02 pH units. Voltages between -2.5 V and +2.5 V can be measured with 0.6 mV resolution. A current follower circuit provides current-to-voltage conversion for detectors which provide a current output. Currents can be measured on four auto-selecting scales between +/- 2.5 mA, with 0.6 nanoampere resolution at the lowest scale. A voltage follower isolates the DAC, which provides a software-programmable voltage output to operate sensors such as thermistors. The DAC can provide voltages between -2.5 and +2.5 Volts in 20 mV increments (the newest version of the hardware has 1 mV increments). A multiplexer allows software selection of the signal source applied to the analog-to-digital converter (ADC). The ADC provides a digital number proportional to the voltage applied. The ADC operates in the range of -2.5 to +2.5 volts. It is a 13-bit ADC, capable of resolution to one part in 8192. (Note: gameport devices are 8-bit, with resolution of one part in 256). The counting circuits are capable of registering 65,535 counts. There are four quartz-based timers with 100 microsecond precision, capable of timing up to 298 days. Push-buttons and switches are also available to control experiments (73,75).

The MSU lab interface has very visible inputs and outputs; it is not a "black box" to the student. Color-coded plugs make it easy to differentiate inputs and outputs. Red LEDs light up when a particular input or output is being accessed by the computer. The design of the interface box gives the user the opportunity to "see" what is going on in the interface box during the experiment. This is where the process of science fits into the hardware design. Given the wide variety of inputs and outputs, the student has the freedom to make several different measurements at the same time. With the software, the students can take the measurements they want when they want, while storing or displaying data in the manner they choose. *The students can design the experiment!* The computer simply acts as an extremely patient lab partner which

will do what the student tells it to do. The computer can capture events that happen too quickly for humans to observe quantitatively, or wait indefinitely for events to happen (77). Unlike a human lab partner, it will not miss a measurement because it wasn't paying attention! As you will see in the next section, it is the software which provides the crucial link between the capabilities of the interface box and its usability by the student. It is the software which truly makes the computer a lab partner.

Software

One of the most important factors to consider when applying computers in a laboratory course is ease-of-use. Students and instructors using the lab interface system must find the software easy to use. The users must feel that *they* have control over the experiment, not visa versa! The software should allow a student to concentrate on the experiment rather than on computer programming.

The software which allows the user to operate the MSU lab interface hardware can be referred to as the *user interface* for the instrument. I will use the terms *user interface* and *software* interchangeably. Barnett states that a user interface is "the collection of keys, displays, screen dialogs, printed output, and protocols with which [the user] interact" (78). Barnett also says that designing a user interface is "more like writing and directing a movie than planning a scientific experiment" (78). Designing the software for a user interface can be more of an art than a science. A well designed and organized user interface will effectively communicate the intentions of the designer to the user.

Design Principles

Reference 78 provides an excellent overview of the principles behind designing a user interface. These basic principles have been followed in the design of the MSU lab interface software and continue to be re-evaluated as revisions to the

software are made. The following is a summary of some of the important design principles from reference 78:

-- *The first principle: know the user!* We must assume that our users, the students, have no background in computers and very little background in chemistry. This led to the development of a system of menu-driven programming for the interface. This also led to the development of a tutorial book specifically designed to assist the students in learning how to use the interface (79).

-- *The user interface should be easy to learn.* Knowing what needs to be accomplished in an experiment, choosing the most important functions should be obvious. Again, this was accomplished with menu-driven programming and the use of function key commands.

-- *The software should be invisible.* The students must be allowed to concentrate on the process of science and experimentation rather than on the software.

-- *The software should minimize memorization.* Most students use the software only once a week. Commands should be selected, not entered. Help screens and messages should be available regarding selections. Yet again, menu-driven programming provides the means for adhering to this principle.

-- *The user interface should be well structured.* The tasks required in using the interface should be broken down into small, logical chunks. The software must not seem like a randomly generated morass. We have designed the software to emulate the process of science. The chunks represent the steps in the process of science discussed in Chapter 3. A well organized software package will also allow for the rapid execution of common operations.

-- *Engineer for errors!* This is one of the most important principles and probably one of the most difficult to incorporate in the initial design stage. The users should *like* to work with the software. It should be forgiving, allowing reversible decisions. The common user errors *must* be engineered out of the system. When

errors do occur, good error messages should appear which lead the user to a solution. We have constantly updated the software to eliminate common student errors.

The MSU Lab Interface Software

Adhering to the principles discussed above, the MSU lab interface software was designed using a menu-driven, English language programming package. A powerful programming language was needed to develop a user interface with the flexibility of this package. The entire software package was written in the C programming language. The C language was chosen for its versatility, modularity, and compactness of code. It allows virtually unrestricted access to the computer hardware (71).

The MSU laboratory interface software consists of five major sections: Main Menu (also known as the Driver), Calibration, Builder, Runner, and Spreadsheet. Figure 11 shows the organization of the software. The phrases in parentheses in Figure 11 are the wording used by the main menu to describe the function of each section to the user.
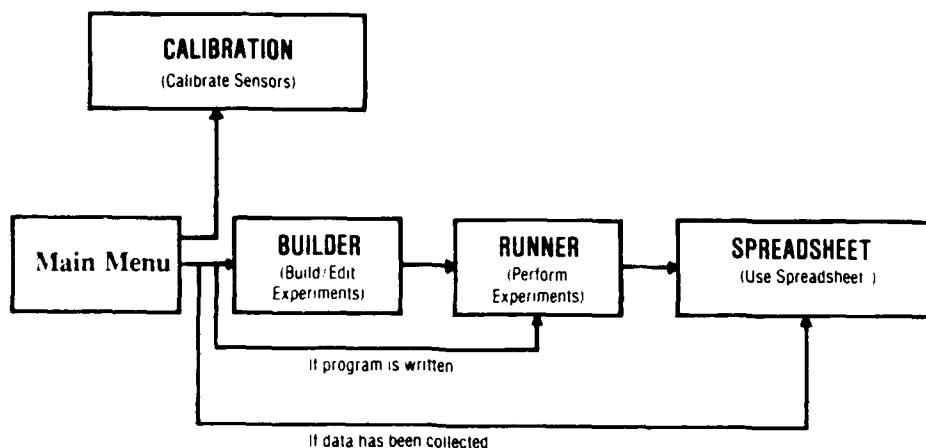
FIGURE 11. MSU Lab Interface Software Flowchart (79).

Note that the flow of the software in Figure 11 is designed to *replicate the process of science* discussed in Chapter 3. The **Builder** allows the user to *design the experiment*. The **Runner** performs the experiment and *collects data*. Finally, the

Spreadsheet allows the user to graph and analyze the data, assisting the user in *organizing the data and drawing conclusions.* I will only briefly describe these sections in this thesis. Each section is described in detail in reference 76.

Main Menu. The **Main Menu** controls the other four software sections. The **Main Menu** binds **Calibration, Builder, Runner,** and **Spreadsheet** together. As shown in Figure 12, the **Main Menu** appears as a menu from which the other components of the software can be selected.
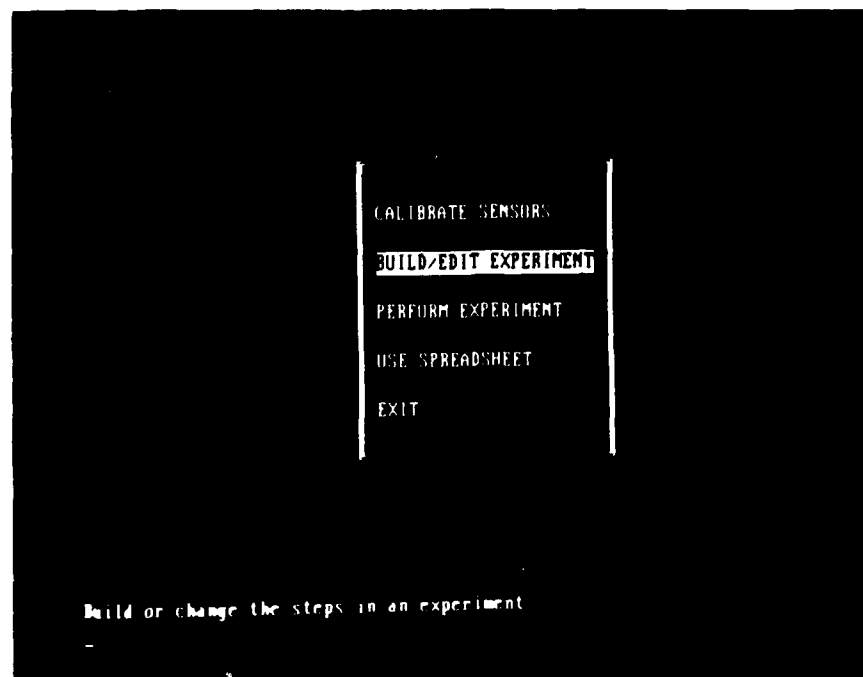
FIGURE 12. Screen Photo of the **Main Menu** Options.

Calibration. The **Calibration** subprogram allows the user to calibrate thermistors or pH electrodes which are attached to the interface box. The thermistors use a two-point temperature calibration procedure, whereas the pH electrode uses a one-point calibration. The screen instructions are self-explanatory, guiding the user through the necessary steps for a proper calibration.

Builder. The **Builder** is the unique core to the entire software package. The **Builder** allows the user to design programs to control experiments. This is the key portion of the software which actively involves students in *experiment design*. Programs can be built from scratch and easily edited using menus in the **Builder** mode. All of the commands available to acquire data or control the experiment via the lab interface are presented in a menu. Figure 13 shows a program created with the **Builder**, along with the menu structure for the commands. This is a simple example of a data acquisition program. The topic of designing these experiments will be developed further in Chapter 5.
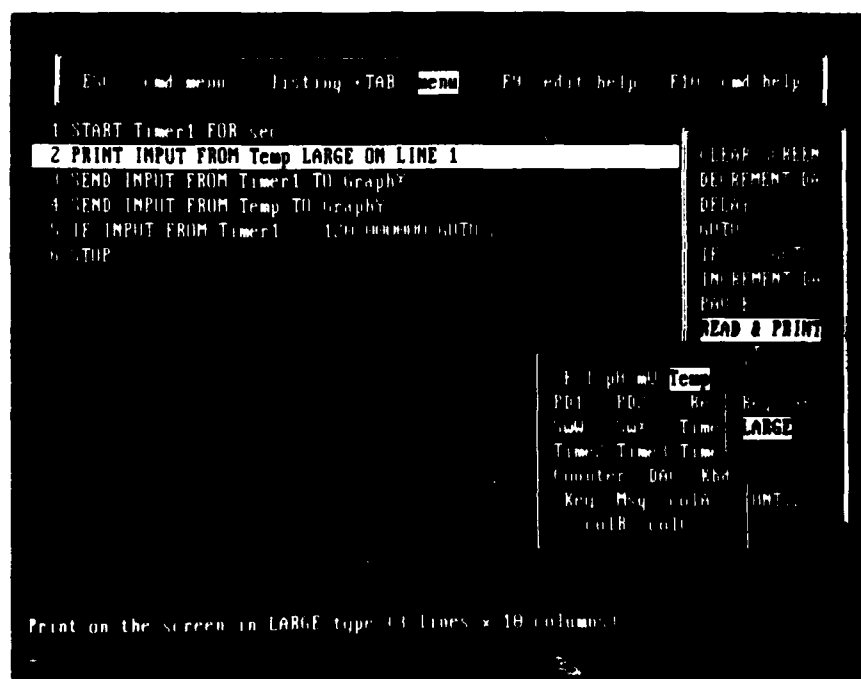
FIGURE 13. The Menu Structure of the **Builder** Subprogram.
Note the many possible sensor inputs.

There are 15 simple commands available to the user. These commands are not entered from the keyboard as in conventional programming languages. All of the parameters and information needed for a certain command are also chosen from sub-menus appearing after the command is selected. This menu-driven structure virtually

eliminates the occurrence of "syntax" errors, a common frustration when programming in a language such as BASIC. The software creates each programming line based on the student's menu selections. Any program "written" by the student will run; it may not run as intended, but it will not "crash." This reduced margin for error decreases the amount of time required for "debugging" an experimental program, freeing time for the student to experiment with different methods. All of the menu-driven portions of the **Builder** display helpful messages and/or examples on the bottom two lines of the screen.

The student can design programs which will actively graph data on the screen during the experiment. Programs can also be designed which send data to up to three spreadsheet data columns for later analysis. Once a program has been created, it can be saved on the student's own floppy disk.

The flexibility of the menu-driven command system allows for a tremendous amount of creativity. Students can create a unique experiment program, taking data in a manner *they* determine. Even if an entire laboratory room is working on the same experiment, the software individualizes the task. Students are no longer simply hooking up an instrument to an experiment. They are *designing the experiment*!

Runner. Once the experiment program has been created in the **Builder**, the **Runner** allows the user to perform (or run) the experiment. After specifying the name of the experiment file to run, the user has several selections with respect to how the experimental data will appear on the monitor. The user can simply display specified numerical data, have the data appear in a spreadsheet column format, or display a graph on the top or right side of the monitor. The **Runner** can use information coming directly from the interface box, or from a simulated experiment data file. The **Runner** *collects the data* in exactly the manner specified by the student.

Spreadsheet. After the experiment has been performed, the **Spreadsheet** option can be used to mathematically analyze and graph the data. The **Spreadsheet** is

a data analysis package, useful for the wide variety of data obtained in laboratory experiments. We established the following "wish list" for the design of a data analysis package (80):

(1) *The data analysis package must be easy to use!* Every other "wish" that follows also revolves around this point! We are totally convinced that commercial spreadsheets, as data analysis packages, are *not* easy to use, at least if you want to use any portion of the lab period for something else besides learning how to use the spreadsheet. Commercial spreadsheets are extremely versatile, which makes them inherently difficult to use. A data analysis package must walk the fine line between versatility and ease of use.

(2) *The data analysis package must support a wide variety of calculations.* When analyzing data, a student must be able to write a formula, just like with a pencil and paper, and be done. The package must have the capability to perform all trigonometric functions, logarithms, derivatives, integrals, and all basic mathematical functions with very logical commands.

(3) *The student should be able to view the raw data and calculations simultaneously.* This allows the student to very easily see the relationships between mathematical operations and the raw data. In addition, the student must not lose the raw data after performing a calculation with it.

(4) *It must be easy to convert data to graphical form.* Simply specifying a given set of data or calculated values to an axis should be enough to generate a graph.

The menu-driven **Spreadsheet** data analysis package incorporates all of the items on this "wish list." Figure 14 shows the appearance of the **Spreadsheet** on the monitor screen. The menu items across the top of the spreadsheet are the available functions and support the process of science discussed in Chapter 3. Columns A, B, and C are available for storing data obtained from the lab interface box. These columns can also be used to input data via the keyboard. Columns D, E, and F are data analysis columns. These columns are used to perform mathematical operations on

information in any of the six columns. The information in the columns can be graphed simply by selecting a column to represent the x-axis and other columns to represent one or two y-axes. A linear regression option makes it possible to obtain a best fit straight line for the data. Statistical information can also be found for each data column. All of the information displayed on the monitor can also be printed out. The rapid calculational and graphing capabilities of the **Spreadsheet** remove the tedium of repetitious calculations and time consuming graphs. Some examples of calculations and graphs obtained with the **Spreadsheet** will be shown in Chapter 6.
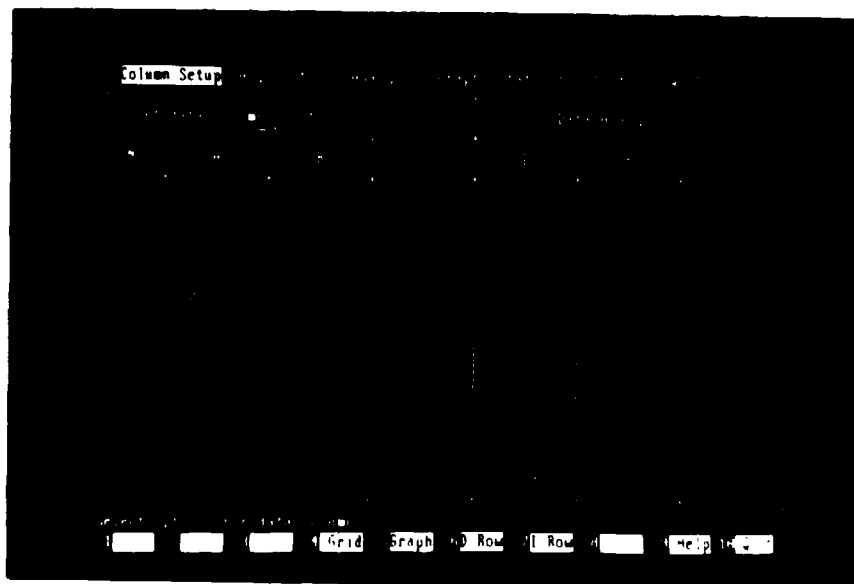
FIGURE 14. The Spreadsheet.
Note that the process of science is followed in the command header.

Through our experience, it is clear that a powerful, easy-to-use spreadsheet provides one of the most useful functions for a computer in the chemistry laboratory. The **Spreadsheet** can divert an enormous amount of time from boring, repetitious tasks

to exciting, thought-intensive tasks. Students can discover key chemical concepts by exploring simple mathematical relationships and graphing experimental data. As Fuller puts it, a spreadsheet "may be used as an empirical 'function finder'" (81). By guessing a mathematical relationship (e.g. exponential, square, inverse), a student can use a formula in the **Spreadsheet** and compare their experimental data to the guess. Graphing and exploring functional relationships makes the **Spreadsheet** a perfect tool for *organizing data and drawing conclusions from that data.*

The MSU laboratory interface system is an integrated hardware/software system designed to involve the student in the process of science. The hardware is designed to make a variety of measurements with research precision, while the software is designed to give the student total control over the system. In the next chapter, I will discuss how the MSU laboratory interface system has been integrated into the chemistry laboratory.

CHAPTER 5

THE LABORATORY PROGRAM

> The challenge of doing science grows out of human beings'
> interest in trying to explain nature in an orderly, systematic way.
> We do not want to be told about nature by other people, but to
> discover it for ourselves. To be true to this spirit, we have to
> give the student the opportunity to be a scientist, to do some kind
> of science. . . . Our crucial task is to put students in a situation
> where they start interacting, not just with the professor's ideas,
> but with what really happens in the world, and to require them to
> construct understandings for themselves. The students have to be
> actively involved in trying to figure things out for themselves,
> not just writing down memorized rules that you told them or that
> they read in a book (81).

The above quote by Dr Robert G. Fuller, a leader in promoting the use of computers to do empirical physics, beautifully and concisely states what we are trying to accomplish in our laboratory program with the introduction of computers. As I have stated many times already in this thesis, we want to use computers in the laboratory to help teach the *process of science*. The MSU lab interface system provides a tool by which we can put discovery-type exercises into the lab. The enormous capability and flexibility of the system creates the opportunity to pose problems to students for which the answers are not already written down and the procedures are not already defined. In addition, students get an opportunity to experience the application of computers in chemistry and some of the basic aspects of computer interfacing.

There have been several efforts to incorporate the teaching of computer interfacing in undergraduate laboratories (82-84). Undoubtedly, there have been many other undocumented efforts. These efforts have been incorporated in a small scale for more advanced students, including physical chemistry or honors classes. The courses usually require programming knowledge in BASIC and develop skills in

computer architecture and electronics design. At Montana State University, we are the first to apply the microcomputer-based laboratory (MBL) concept on large scale to all introductory chemistry students, science and non-science majors alike. Unlike other efforts, we de-emphasize programming language and electronics knowledge. We emphasize computer interfacing as a laboratory tool, not as a course in itself. We make the computer as much a part of the lab as beakers, test tubes, and chemicals.

In this chapter, I will detail the incorporation of computers into the laboratory program at Montana State University. I will begin a discussion of the workshops which led to the method for developing students' computer skills. I will then describe a pilot program which led to large scale implementation, followed by a discussion of the integration of the computer into the entire general chemistry program.

Workshops: The Foundation

A series of workshops, from November 1988 to June 1989, provided a proving ground for many of the concepts which we incorporated into our chemistry laboratory program. These workshops, along with several shorter demonstrations, provided training to teachers and scientists on how to use the MSU laboratory interface. Three workshops in particular were aimed at Montana science teachers. During these three two-and-a-half-day workshops, sponsored by the Montana Board of Regents and Montana Office of Public Instruction, we trained teachers on how to effectively design experiments using the MSU lab interface system. Each teacher received an interface system to take back to their schools. A total of 76 junior and senior high school science teachers from throughout the state of Montana participated in these three workshops. These workshops have been described in reference 7? Later workshops expanded this number of teachers to 106 by April 1990. These workshops were very important to the success of our laboratory program because they provided a "laboratory" in which to test and evaluate our ideas on a very compressed time frame. The short turn-around time of these workshops provided us with rapid feedback with

respect to what worked and what didn't work when teaching others how to use the MSU lab interface system. Since the computer component of our laboratory course grew out of our workshop experience, each workshop provided the equivalent of a classroom quarter of feedback compressed into two and one-half days.

We started the workshops with the assumption that the participants had no computer experience. In reality, only a handful of teachers had any previous experience in programming. This reaffirmed our belief that most teachers do not have the time or background in traditional programming languages (e.g. BASIC) to write good computer interfacing software. We realized at this time that as we developed our chemistry laboratory course, we must assume that our students would not be computer literate.

The goal of each workshop was to have each teacher leave with the confidence and experience to design their own laboratory experiments using the MSU lab interface system. (This is precisely the level we wished to achieve with our chemistry students.) To accomplish this goal, our workshops included:

        (1) instructor demonstrations,

        (2) instructor-led experiment designing exercises,

        (3) teachers performing staff-designed experiments, and

        (4) teachers designing experiments.

The last three steps proved to be the general structure we used to incorporate the MBL concept into our chemistry labs, substituting the teachers' role with students.

Instructor Demonstrations

As an overview of the system, we first demonstrated its capabilities to the teachers. In this introductory phase of the workshops, we designed and built experiments using a thermistor probe and a colorimeter as representative of the types of experiments which can be done with the interface. During these demonstrations, we showed how quickly and easily an experienced user could design and perform

experiments. In addition, we showed the data analysis and graphing capabilities of the **Spreadsheet**. In showing the teachers what could be done, we hoped to get them thinking about how they could apply computer interfacing to their own laboratories. Although extremely useful at the workshops, these instructor demonstrations were left out when the computers were incorporated into our chemistry laboratories, due to lack of time.

Instructor-Led Experiment Design Exercises

We established early in the workshops that designing experiments required little or no programming experience. The teacher must not have to wade through volumes of computer manuals just to get the computer to perform a simple task. We guided the teachers through the design of a simple experiment which allowed a thermistor probe to display a temperature on a monitor screen. The simple, four line program shown in Figure 15 illustrated the ease-of-use of menu-driven programming to perform a task. We used this temperature experiment to illustrate the programming steps which must be made when making a measurement. We later incorporated this same exercise into our student chemistry labs.

```
1 PRINT INPUT FROM Temp LARGE ON LINE 2
2 IF INPUT FROM SwX = On GOTO 4
3 GOTO 1
4 STOP
```

FIGURE 15. Simple Program to Measure Temperature
and Display the Value on the Screen.

Once they completed the temperature experiment, the teachers designed an analogous experiment using a cadmium sulfide (CdS) photocell to measure light intensity in the room. We then guided them through modifications of their photocell experiment which allowed graphical displays and data storage and analysis. The

photocell is an excellent demonstration tool for lab interfacing because of its responsiveness and wide applicability in the science lab. A bare CdS cell (see Figure 16) has no hidden surprises or black box aura; students and teachers are not intimidated nor confused about what the cell is doing once the basic operation is explained. The CdS cell learning exercises proved so successful as a teaching tool at the workshops that the same procedure was adapted for our chemistry lab courses.

FIGURE 16. A Cadmium Sulfide (CdS) Photocell.

Performing Staff-Designed Experiments

Once the teachers learned the basics of interfacing and programming with the MSU lab interface, they performed a number of staff-designed, or "canned", experiments. This segment of the workshops had two purposes. First, the teachers looked at the programs and saw some of the different menu-selected programming commands used depending on the level of sophistication desired. This was done as a group and individually. Secondly, in performing these "canned" experiments, the teachers saw how previously designed experiments could be modified to perform an

experiment and obtain data in exactly the manner they wish. "Doing" was the key in these workshops. Performing experiments simply gave the teachers more experience with the interface system. Making the workshop an active experience was very important!

In addition to the reasons just cited, the "canned" experiments also played an important role in our general chemistry classes. During some of the early laboratories of the quarter, students did not yet have the skills necessary to design the programs needed to perform the experiment. The "canned" experiment programs allowed them to use the interface as a tool even though they did not yet have skills to program it themselves. In essence, students can still use the interface as an instrument in the lab even while they are learning the nuts and bolts.

Teachers Designing Experiments

The most important part of the workshop arrived when the teachers were allowed to design their own, individual experiments. Based on their own ideas and what they had seen in the workshop, teachers designed and built experiments using the lab interface which applied to their science discipline and their laboratories.

The results of these teacher-designed experiments were truly displays of creative science! Examples of experiments designed and tested during the workshops included pressure-sensing wind tunnel measurements, gun powder burn rate experiments, carbon dioxide exhalation monitoring, stream turbidity measurement, photosynthesis monitoring, pendulum timing, thermometric titrations, reaction time experiments, and *many* others. Most of the teacher-designed experiments simply could not have been performed with their current, conventional laboratory equipment. More importantly, teachers were placed in the *experiment design* mode, not reliant on pre-defined experiments. The teachers also recognized (as did we!) that students can

be placed in the same experiment design mode using the lab interface system. At the end of the workshops, the teachers demonstrated their experiments to the other workshop participants.

These workshops and several others laid the foundation for integrating the computers into our chemistry laboratories. We learned what needed to be taught to beginners and how much time it would take to do it. It *was possible* to teach others to use the MSU lab interface for experiment design in a relatively short amount of time. Using what we had learned at the workshops, we initiated a test program in which one section of Chem 125, Introductory Chemistry Laboratory, would design their own experiments using the MSU lab interface system during Winter quarter 1989.

A Pilot Program: The Framework

To evaluate the concepts developed in our workshops, we applied our process of science approach to using computers in the laboratory to 22 randomly selected students in Chem 125, beginning in January 1989. Besides this thesis, a discussion of this pilot project can be found in reference 74. Chem 125 is the introductory chemistry laboratory designed for non-science majors, or for students using the course as a stepping stone to the more advanced Chem 130 series.

The 22 students in this "experimental" section had a wide variety of backgrounds, which is typical of Chem 125 students in general. Of the 22 students, 18 (82%) had taken a high school chemistry class. Only 27% of the students had taken even a beginning level calculus class (this percentage would have undoubtedly been smaller had this been a group of Fall quarter students). For 32% of the students, their highest level of math was a college trigonometry course. The remaining 41% had either taken a class in business math or basic algebra. Two-thirds of the students had taken a computer course in high school or college. Almost all who had computer experience said that it was in the area of word processing, not programming. It is

reasonable to assume, then, that the students have only minimal math and computer skills when entering Chem 125. I assumed that the students had absolutely *no* computer skills.

The experimental course was designed to be completed in a nine week (Winter and Spring) or ten week (Autumn) laboratory quarter. Each laboratory period was three hours. Because of one week lost for cold weather in February, only eight laboratory periods were available for Chem 125 during the Winter quarter. As a result, some shuffling occurred with respect to the lesson schedule. Since the lesson content so closely resembles that for the full scale lab program, I will not discuss any details of the lessons until the next section of this thesis.

The first five weeks (one lab period per week) were spent doing "conventional" laboratories, such as thermochemistry, spectroscopy, and qualitative analysis. Students used "canned" interface programs in these experiments to acquire data when appropriate. In addition, students spent about one hour during each of the first five lab periods learning some of the basics of using and programming the MSU lab interface. During the last three periods, students chose a chemical problem and were required to design an experiment using the MSU lab interface to collect and to analyze the data. This final project, as it turns out, was the key to involving the students in the process of science with the help of the computer.

As with the teachers, menu-driven, user-friendly programming greatly facilitates the application of laboratory interfacing for students. However, the student must have a certain amount of "computer logic" or "programming logic" to design an experiment with the interface system. The specifics of how this is taught will be covered in the next section. In a general sense, to develop experiment skills using the MSU lab interface, we do the following: 1) teach a small amount of computer basics, 2) familiarize the students with the structure and sequencing of the interface software, (3) guide students through short, easy-to-write experiments, and 4) let students use more complex "canned" programs to do experiments and let them examine the

structure of these programs. This approach proved to be extremely successful in the pilot section and was adapted for all Chem 125 and Chem 135 (General Chemistry Laboratory) in the Autumn 1989 quarter. The proof of success came during the final projects.

The rationale behind the final project concept is quite simple. Let students choose an experimental problem of interest with a set of objectives. They then use the *process of science* to accomplish the objectives. Students in the Chem 125 test section chose an experiment project which they were to carry out during the last three weeks of the quarter. For the projects, they were required to obtain and to analyze their data using the MSU lab interface system. These projects included a variety of acid/base titrations on household chemicals and medicines using a pH electrode. In addition, there were other projects involving colorimetry, electrochemistry, and turbidity measurements. The students were given a short description of their experiment. The description included some background information, the type of data they needed to obtain, a small amount of experimental procedure information, and some reporting requirements. In some cases, students needed to use outside references. The objective of the descriptions were to give students enough background to get them started, but not enough background to tell them exactly what to do. Essentially, I wanted to take them away from the "cookbook" mode and place them in the "experimental" mode.

Given this information the students, working with a lab partner, then needed to decide how to configure the interface to get their data. Within three hours of the nine hours available for the projects, *every* pair of students had properly configured the interface and had a functional program to acquire their data; some were ready within an hour. The remaining six hours were then available to do experimental work.

Because of the speed and ease-of-use of the lab interface system, students were able to repeat their measurements many times and to try different procedures-- that is, experiment! All groups were required to hand in tables and graphs of their data with their report, along with a listing of the program which acquired their data.

56

Figure 17 shows a Beer's Law plot of absorbance versus concentration using student-made standards of Cu^{2+} solutions. The students used a home-made colorimeter attached to the interface to obtain their data. They then used the **Spreadsheet** to convert the light transmission data into an absorbance and to graph the data. Students then used their graphs to find unknown concentrations of Cu^{2+}. Some students produced pH titration curves, voltage vs time curves, current vs time curves, and other graphs. The final reports on the projects showed that the students had a very good grasp of how to design an experiment to acquire the data required to solve the experimental problem. In other words, the students were able to employ the computer to help them use the scientific method. They were able to get the interface system to do what *they* wanted it to do.
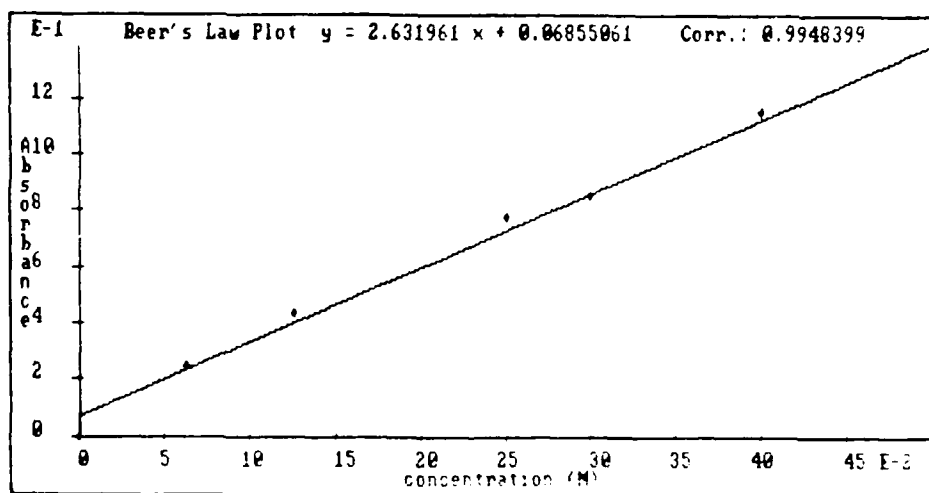
FIGURE 17. Student Graph Illustrating Beer's Law for Cu^{2+} solutions.

In our pilot program, we successfully taught introductory chemistry students to use computers in the process of science in a minimal amount of time. In a post-course survey, students indicated that they like having computers in the lab. They could see the usefulness of the lab interface system to make measurements and to analyze data. More important, students felt that the final project challenged them and that they

learned a great deal from it. In addition, many of the students indicated that they subsequently felt more comfortable around computers in general. We proved that the lab interface system *can* be used successfully by a wide variety of students. It was apparent that involving the students in the process of science, especially in experiment design, greatly enhanced the students' laboratory experience. Based on these results, we incorporated this laboratory concepts into *all* of the introductory and general laboratory courses beginning in the Autumn 1989 quarter.

Going Full Scale: Involving All of the Students

It was the original intent of this project to develop a course for Chem 125 (non-science majors) only. In the summer of 1989, the decision was made to expand the MBL-final project concept into the Chem 135/Chem 136 (science and engineering majors) sequence as well, beginning in the Autumn 1989 quarter. In this section of my thesis, I will describe the content of these courses and the approach taken in their development.

The Overall Approach

The overall approach to the Chem 125/135 courses was nearly identical to that for the Chem 125 pilot section. The first six or seven lab periods would be used to develop *conceptual, lab,* and *computer skills* (see Figure 18). These skills would be tested in the final project during the last three weeks of the quarter. As much as possible, the conceptual, lab, and computer skills learned during any particular period blended together and supported each other, while building in complexity each week as these skills improved.

Integrating the development of conceptual skills with the development of lab and computer skills proved to be the most difficult aspect of designing the Chem 125/135 courses. Not only must laboratories be chosen which build upon a student's knowledge of basic chemical concepts, but they must also be chosen to be

Concepts

Lab Skills

Computer Skills

Final Projects
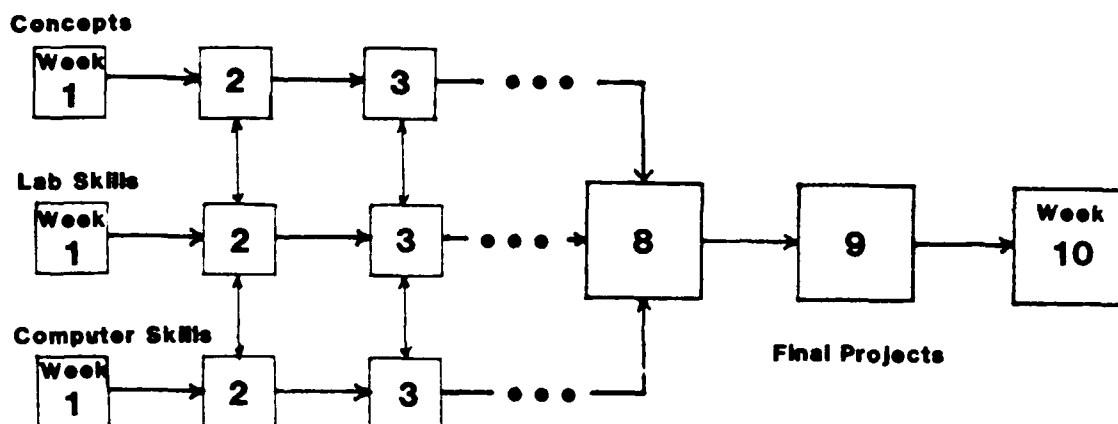
Week #	Concept	Lab Skill	Computer Skill
1	Density of liquids, solids, and solutions	Use of a balance, volumetric glassware	Making simple graphs with the Spreadsheet
2	Heat loss and heat gain, heat from chemical and physical changes	Operation of a Bunsen burner, using a thermistor to measure temperature	Design an experiment program to measure temperature
3	Periodic trends in the elements	Develop observation skills, measure conductivity	Design an experiment to make light intensity, current measurements
4	Interpret emission spectra, understand Bohr model of atom	Construct and calibrate a spectroscope, observe spectra	Change data displays for light measurement, make calibration graph
5	Write, balance, and predict chemical reactions and formulas	Perform "spot test" reactions, separate ions in solution	Get information to data file which interface cannot measure
6	Use mole concept to determine amount and % element in unknown	Separate, weigh reaction products, vacuum filtration, dehydration	Additional practice and programming tips
7	Acids, bases. strong vs weak acids, pH, buffers, acid rain	Use of pH electrode and indicators, micro-techniques	Design an experiment to measure pH, math. analysis techniques, more graphing

FIGURE 18. Flowchart for the Introductory Chemistry Laboratory, Chem 125.

complementary to the material being presented in the lecture class. Obviously, the
labs must also be chosen so that students have all the skills necessary to successfully
complete the final projects. Considerable coarse and fine tuning still must be done so
that the development of all of these skills flows smoothly.

From an instructional standpoint, we decided to put the development of
computer skills into a separate book (79). We believed that the lab interface would be
easier to learn to use if a separate reference was available. Thus, students had a
conventional laboratory book and a computer book for Chem 125/135. Without
question, however, the key for the development of the students' skills is the laboratory
instructor, or teaching assistant. With this fact in mind, we developed a laboratory
instructors' training course for the MSU laboratory interface.

Laboratory Instructor Training

It is essential to give laboratory instructors adequate training so that they feel
comfortable with their responsibilities and are competent at what they are doing.
Since our approach in the general chemistry laboratory was radically different from
previous years, we developed a four-day laboratory instructor training workshop. The
general approach toward the workshop was to take the instructors through exactly the
same exercises as they would be expected to teach their students, with emphasis on
identifying potential problems and pitfalls. This inaugural training workshop was held
from 5 to 8 September 1989.

We began the workshop with a demonstration of the MSU lab interface. The
purpose of the demonstration was to simply give the lab instructors a glimpse of what
the system could do. We then reviewed the basic instrumentation aspects of the lab
interface. The purpose of the instrumentation review was to stress that it is important
for the instructor to let their students know that the lab interface must be viewed as a
multi-purpose instrument. Everything that we teach the students about the lab
interface system supports their using the interface as an instrument in the laboratory.

We followed the lab interface basics by having the lab instructors design a simple temperature experiment. The instructors would be teaching the same procedure to their students the second week of the quarter. The purpose of this exercise was make all of the instructors comfortable the lab interface system.

Beginning the second day of the workshop, we placed the lab instructors in teacher/student role playing situations. In one exercise, one of the lab instructors taught their partner how to make the lab interface function as a pH probe, using the procedure they learned the previous day with the temperature probe. Switching roles, the other lab instructor taught his or her partner how to design an experiment which measured voltage. The purpose of these role playing exercises was to emphasize to the lab instructors that they must be able to *show* and *explain* to students how to do things on the interface system and *not* do it for them. In addition, the lab instructor must learn to trouble-shoot experiment programs for students, recognizing what the program is *supposed* to do and determine what must be done to make the program work. These role playing exercises were followed by the examination of "canned" programs, which gave the lab instructors practice in determining and explaining the function of various programs which students would be using and designing.

After learning the interface basics, we taught each lesson from the computer book (which will be discussed in detail in the next section) as the instructors would with their students. Paralleling these lessons, we allowed the lab instructors to perform the Chem 25/135 labs which they would actually be teaching during the quarter. There is no substitute for actually doing a laboratory to obtain a full understanding for the concepts being taught in the lab, as well as to identify potential problem areas for students. The teaching of computer lessons and performing of laboratory experiments occupied roughly three-fourths of the total workshop time.

Dispersed between the previous workshop exercises, we included various other information sessions. We discussed laboratory safety, chemical waste handling, grading, drill and tutorial software, and general laboratory procedures. In addition,

we had feedback sessions at the end of each day to discuss possible improvements in the laboratories and the lab interface hardware/software package. These sessions were crucial toward developing a laboratory program which would be supported by the lab instructors and run smoothly in the Autumn quarter.

As I have detailed thus far in Chapter 5, considerable background work went into establishing our laboratory program for the Autumn 1989 quarter. The workshops, pilot program, and laboratory instructor training sessions all provided important information for developing our approach to the general chemistry laboratories. The most tangible results of this development were the *computer book* and *laboratory manuals* for the Chem 125/135/136 courses.

The Computer Book

An important part of this research project and the laboratory program in general was to develop a book which served as a guide for students toward developing the necessary computer skills in the laboratory. This book, titled *Using Computers in the General Chemistry Laboratory* (79), was designed to build confidence and competence in using the computer on a step-by-step basis. The book was designed to be a *guide*, not a self-contained tutorial. Therefore, the lab instructor played a key role in clarifying the computer skills presented in the book. Because of the length of the manuscript, I will not reproduce this book as part of this thesis. The manuscript is readily available through the Department of Chemistry or the MSU bookstore. Instead, I will discuss the overall content of the book, emphasizing the highlights. The table of contents of the book is shown in Table 1.

Lesson 1: Getting Started. Lesson 1 serves as a "get acquainted" lesson for the students. The lesson includes introductory material about the computers, a discussion of basic disk operating system (DOS) commands, and an overview of the structure of the MSU lab interface software. To gain confidence with the menu-driven software, students complete lesson 1 with a simple graphing exercise using the **Spreadsheet**. The

Table 1. Table of Contents for *Using Computers in the in the General Chemistry Laboratory.*

Lesson #1:   Getting started: Some basic disk operating system commands

Introduction to the lab interface software

A graphing and **Spreadsheet** exercise

Pre-quiz

Lesson #2:   The MSU lab interface as an instrument

Experiment design: Building a temperature probe experiment

Pre-quiz

Lesson #3:   The photocell as an instrument

Building a photocell experiment (Part 1):
    A basic measurement

Pre-quiz

Lesson #4:   Building a photocell experiment (Part 2):
    Changing how you display data

Pre-quiz

Lesson #5:   Sending keyboard values to a **Spreadsheet** file

Pre-quiz

Lesson #6:   More practice and programming tips

Pre-quiz

Lesson #7:   More graphing and math functions

Appendix: Calibrating thermistors and pH probes

A tutorial: Keystroke usage when building an experiment

students construct a Celsius-to-Fahrenheit conversion graph and obtain the conversion formula by doing a linear regression analysis.

Lesson 2: Designing a Basic Experiment. Lesson 2 begins with an overview of how the MSU lab interface and the computer serve as components of an instrument system. The lesson continues with the students' first exposure to designing experiments: using a thermistor as a temperature probe. Lesson 2 is probably the most important lesson in the book, since it is here the students learn the four major steps to follow when designing an experiment with the lab interface software:

1. **Make the measurement**

2. **Repeat the measurement (loop)**

3. **Exit the measurement loop**

4. **Make the program friendly**

As an example of this strategy, students build a variety of programs centered around using a thermistor as a thermometer. When designing an experiment, you must build your program around the measurement you wish to make. These are the lines which the rest of the program will support. *Making the measurement* may take only one or two lines of the program. For example, if you wanted to display a temperature from a thermistor on the monitor screen, the command line

```
1 PRINT INPUT FROM Temp1 LARGE ON LINE 2
```

would accomplish the task. This one-line program would read the temperature from the thermistor input (Temp1) and display large numbers in the middle of the screen (LARGE ON LINE 2). However, it would only do this once and the program would stop. The program would have to be re-run to get a new temperature.

The students quickly realize that this one-line program, though functional, is not much of a thermometer. For most applications, you would want a measurement

displayed more than once. To use the thermistor as a true thermometer, you would want the temperature reading updated as many times as possible during a given time interval. Adding a "GOTO" statement to the above program can create a *measurement loop*:

```
1 PRINT INPUT FROM Temp1 LARGE ON LINE 2
2 GOTO 1
```

Line 1 causes the thermistor reading to be displayed on the screen. Line 2 instructs the program to then return to line 1 and display a new temperature. After building this program and running it, students quickly realize that there is nothing in the program which will cause it to stop. It is infinite loop and will continue to loop back to line 1 until the program is aborted.

To *exit the measurement loop*, students are shown a variety of options. The most common way to do this with the lab interface software is by using an "IF . . . GOTO" statement, also known as a conditional statement. Students find that one of the best methods of exiting a measurement loop is with the use of external switches on the interface box. These push button or toggle switches are easy to use and are easy for students to logically implement into their programs. The following program now allows the thermistor to operate as a thermometer:

```
1 PRINT INPUT FROM Temp1 LARGE ON LINE 2
2 IF INPUT FROM SwX = On GOTO 4
3 GOTO 1
4 STOP
```

This sequence uses a combination of an IF . . . GOTO statement and a GOTO statement. As long as switch X on the interface box is in the down (Off) position, the program will proceed to line 3 and loop back to line 1. Once switch X is flipped up (On), the program will proceed to line 4 (STOP), exiting the loop. Simply by choosing different inputs (voltage, pH, etc.), the student can convert the above program into a

volt meter, a pH meter, or any measuring device they need for the experiment. The above program provides the basic structure for every measurement the students need to make in the laboratory.

Print statements can be added to make the previous program *user-friendly*. These user-friendly statements either tell the user what's going on or what to do, or they package and display information for the user. The user-friendly statements are important to master in the experiment design process.

```
1 PRINT "This program displays temperature in Celsius." ON LINE 18
2 PRINT "Flip up the x-switch to stop the program." ON LINE 20
3 PRINT "TEMPERATURE" LARGE ON LINE 1
4 PRINT INPUT FROM Temp1 LARGE ON LINE 2
5 IF INPUT FROM SwX = On GOTO 7
6 GOTO 4
7 STOP
```

Lines 1-3 above tell the user what the program does, how to stop the program, and describe the number being displayed. The amount of "user-friendliness" in a program depends upon the person writing the program and for whom he/she is writing it. The user-friendly aspects of experiment design allows students to individualize their experiments.

The four-step experiment design process described here and in Lesson 2 of the computer book provides the central core of what we want the students to learn about using the lab interface software. The remaining lessons focus on controlling how data is obtained, displayed and analyzed.

Lesson 3: Basic Measurements with a Photocell. During Lesson 2, the lab instructor leads the students through step-by-step on how to build the temperature experiment. In lesson 3, the students modify their temperature program to build an analogous experiment using a cadmium sulfide (CdS) photocell. The purpose of this lesson is to show the students that they can make the interface system into nearly any

instrument they need by (1) changing the detector, (2) selecting the proper
inputs/outputs on the interface box, and (3) making appropriate changes in their
software programs. As students discover, only slight modifications to the previous
temperature program are needed to make it operate the photocell.

```
1 SEND 500.000000 TO DAC1
2 PRINT "Flip up the x-switch to stop the program." ON LINE 20
3 PRINT "CURRENT" LARGE ON LINE 1
4 PRINT INPUT FROM I1 LARGE ON LINE 2
5 IF INPUT FROM SwX = On GOTO 7
6 GOTO 4
7 STOP
```

Line 1 of the above program is necessary to send a voltage (500 mV) to **DAC1**
so the photocell will operate. Unlike the thermistor, the photocell is a "dumb" sensor;
it is not internally calibrated with the interface, so it must be "told" exactly what to do.
Rather than reading **Temp1**, the program reads the current (**I1**), which is directly
proportional to the amount of light the photocell receives. Line 4 is still the
measurement line. As you can see, the basic concept of making the measurement
needed for an experiment is quite simple; changing the desired quantity to be
measured is even simpler.

Lesson 4: Changing the Display of Data. Students discover in lesson 4 that
one of the major advantages of the lab interface system is that they can tell the system
exactly *how* they want the information displayed, *when* they want it to take data, and
where they want the data to go. In lesson 4, we show students how to modify their
photocell experiment from lesson 3 so it creates a real-time graph and send data to a
Spreadsheet file. This lesson includes a short, but critical, example of establishing
time as a variable in their experiment. The lines in **bold** type in the following
program show the changes to the previous photocell program needed to (1) establish
time as a variable, (2) plot time on one axis, and (3) plot current on the other axis.

```
1 SEND 500.000000 TO DAC1
2 PRINT "Flip up the x-switch to stop the program." ON LINE 20
3 PRINT "CURRENT" LARGE ON LINE 1
4 START Timer1 FOR sec
5 PRINT INPUT FROM I1 LARGE ON LINE 2
6 SEND INPUT FROM Timer1 TO GraphX
7 SEND INPUT FROM I1 TO GraphY
8 IF INPUT FROM SwX = On GOTO 10
9 GOTO 5
10 STOP
```

Students build the above program, then run it to see what it actually does. One of the advantages of the MSU lab interface system is that you can quickly *design* the experiment you want and rapidly run it to *see* if the experiment does what *you* want it to do.

Making a real-time graph can help students visualize what is happening in an experiment as it's occurring. However, saving data to a file for later analysis may be more beneficial, especially if the data needs to be changed mathematically. The only difference between sending data to a real-time graph, as shown above, and sending data to a **Spreadsheet** file is *where* you send the data. Rather than sending the data to **GraphX** or **GraphY**, you send the data to **COL-A**, **COL-B**, or **COL-C**, which are **Spreadsheet** data columns. Students modify the previous photocell program and send **Timer1** data to **COL-A** of a **Spreadsheet** data file and **I1** to **COL-B** as follows:

```
1 SEND 500.000000 TO DAC1
2 PRINT "Flip up the x-switch to stop the program." ON LINE 20
3 PRINT "CURRENT" LARGE ON LINE 1
4 START Timer1 FOR sec
5 PRINT INPUT FROM I1 LARGE ON LINE 2
6 SEND INPUT FROM Timer1 TO COL-A
7 SEND INPUT FROM I1 TO COL-B
8 IF INPUT FROM SwX = On GOTO 10
9 GOTO 5
10 STOP
```

Again, the lines in **bold** are changes to the previous program. The concept of
making small changes to the experiment design, then *seeing* the result of those
changes is a very effective way of building students' self-confidence in choosing
program design commands on their own. The students learn that the interface can
display numbers on the screen, graph data on the screen, and send data to a file for
subsequent analysis.

Lesson 5: Obtaining Data the Interface Can't Measure. There are many
instances when designing an experiment in which you may want to store a variable
quantity which the MSU lab interface cannot read directly. Examples of such
quantities include solution concentrations, volumes, or masses. The **kbd** (keyboard)
input option allows you to input such variables during an experiment. The **kbd** option
is extremely useful and must be practiced and understood to be used effectively.

To illustrate the use of the **kbd** option, students modify the photocell programs
they built in lessons 3 and 4. As an example, we propose that the photocell is sitting
in a room attached to the MSU lab interface, as shown in Figure 19. The room has
four sets of lights. We want to study the response of the photocell with respect to how
many lights are turned on. In other words, as we throw each light switch, we want to
measure the current from the photocell and store this information. Later, the
Spreadsheet can be used to analyze this "current vs lights-on" data. This example is
totally analogous to relating a pH reading in a titration to a particular volume of acid
or base added to the unknown. The program below accomplishes the task.

```
1 SEND 500.000000 TO DAC1
2 PRINT "ENTER THE NUMBER OF LIGHTS ON" ON LINE 1
3 PRINT "Enter a negative number to stop taking data." ON LINE 2
4 SEND INPUT FROM Kbd TO COL-A
5 IF INPUT FROM COL-A < 0.000000 GOTO 9
6 PRINT INPUT FROM I1 LARGE ON LINE 2
7 SEND INPUT FROM I1 TO COL-B
8 GOTO 2
9 STOP
```
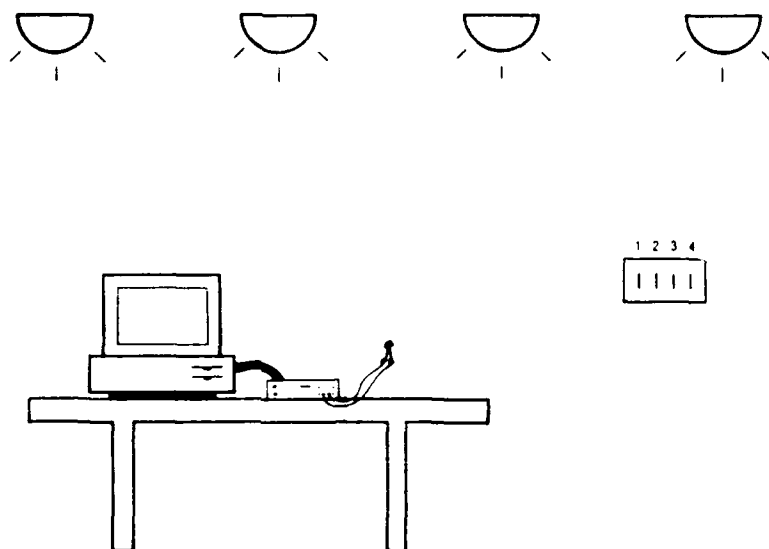
FIGURE 19. Photocell/Lights Experiment (79).

Lesson 6: More Practice and Programming Tips. Students can learn many techniques by which they can "clean up" their experiment programs, making the displayed information clearer and giving them more precise control over when the interface system takes data. During lesson 6, students learn a few of these techniques and have the opportunity to practice these methods. These techniques include pausing the program, waiting until a condition is met, delaying measurement loops, and clearing the screen.

Lesson 7: More Graphing and Math Functions. In preparation for the final projects, students have the opportunity to review some of the graphing and analysis capabilities of the **Spreadsheet**. Students use hypothetical data on the growth rate of two different kinds of bacteria to study log functions, linear regression analysis, and derivatives. The **Spreadsheet** makes previously abstract, but chemically important, mathematical concepts become visible.

The Laboratory Manuals

The laboratory manuals provided the vehicle for learning laboratory skills and chemical concept skills. The laboratory manuals were also designed to allow the students to apply their computer skills to the lab experiments whenever possible.

Two laboratory manuals were developed for the Chem 125/135/136 courses (85,86). Because of their length, the lab manuals are not included as part of this thesis, but copies are readily available through the Department of Chemistry at MSU. Although Chem 125 is an introductory course for non-science majors and Chem 135 is a general course for science majors, we decided to develop one laboratory manual for both courses. The primary reasons for doing this revolved around administrative and logistical considerations. Since this was our first quarter with a new lab concept for 1200 students, it seemed prudent to have all of the lab instructors teaching from the same lab manual. This made it easier for the staff to monitor the labs and help trouble shoot problems. Since most of the labs were completely new or drastically revised, having the same laboratory book for the initial quarter also eased the work load for the chemical preparation staff. We developed a separate lab manual for the Chem 136 course, which is the follow-on to Chem 135.

Chem 125/135. The corresponding lecture courses for Chem 125 and Chem 135 are considerably different. To make the labs correlate with the lectures as much as possible, we made some of the labs different, changed the order that the labs were taught, and added *levels* to most of the labs. As illustrated in Figure 18, both courses consisted of seven "regular" labs from the lab manual, followed by three weeks of final projects. The Chem 125/135 lab manual contained a total of nine different laboratories. Most of the labs contained different *levels*, which allowed us to challenge even the brightest Chem 135 student. The Chem 125 students were normally only required to complete the lowest level, whereas the Chem 135 students were required to complete one or more of the higher levels. For example, in the emission

spectroscopy lab, the first level involved making a calibration graph and viewing unknown spectra. The second level required more exact measurements and complex calculations involving the Bohr model for the hydrogen atom. The level concept allowed us to use many of the same laboratories for both Chem 125 and 135. Table 2 on the following page shows the lab sequence used for Chem 125, while Table 3 shows the lab sequence for Chem 135.

Before the students began the experiments, they spent 30 to 50 minutes on the corresponding computer lesson for that laboratory period (see previous section). Every attempt was made to design the laboratories so that the students would have the opportunity to directly use the computer skills which they learned during the corresponding computer lesson. Because of the teaching methods and examples used in the computer book, this was not always possible. In addition, some of the laboratories (e.g. qualitative analysis and molecular geometry) simply did not lend themselves to be computer interfaced experiments. When a particular experiment required computer interfacing but the students did not yet have the skills to do it themselves (e.g. conductivity of elements), we provided a "canned" program. In short, we did not have a perfect blending of conceptual, laboratory, and computer skills during every lesson. However, we did develop the skills needed to successfully merge these skills during the last three weeks for the final projects. The final projects will be discussed in a later section of this chapter.

Chem 136. With the basic computer skills already developed in Chem 135, it was not necessary to have an accompanying computer book nor to have any specific time designated to improving their computer skills. Students in Chem 136 were expected to retain these computer skills and were required to configure the MSU lab interface to act as a pH meter, a thermometer, a colorimeter, and a volt meter during the seven regular laboratory periods. In addition, they used a canned Geiger counter experiment to perform a nuclear chemistry experiment. To ease logistical conflicts

Table 2. Chem 125 Laboratory Schedule.

Week #1, Experiment 1: Identification of an Unknown Compound by Density
Determination (3 levels)

Week #2, Experiment 2: Flames, Heat, Calories, and Specific Heat (2 levels)

Week #3, Experiment 3: Periodicity: Conductivity of Elements (3 levels)

Week #4, Experiment 4: Emission Spectroscopy: Analysis with Light (2 levels)

Week #5, Experiment 5: Qualitative Analysis: Identification of Unknown Ions
(1 level)

Week #6, Experiment 6: Quantitative Analysis (3 levels)

Week #7, Experiment 7: Acids and Bases (1 level)

Weeks #8-10: Final Projects

Table 3. Chem 135 Laboratory Schedule.

Week #1, Experiment 1: Identification of an Unknown Compound by Density
Determination (3 levels)

Week #2, Experiment 2: Flames, Heat, Calories, and Specific Heat (2 levels)

Week #3, Experiment 6: Quantitative Analysis (3 levels)

Week #4, Experiment 9: Boyle's Law and Charles' Law (1 level)

Week #5, Experiment 4: Emission Spectroscopy: Analysis with Light (2 levels)

Week #6, Experiment 7: Acids and Bases (1 level)

Week #7, Experiment 8: Molecular Geometry (1 level)

Weeks #8-10: Final Projects

with Chem 125/135, the Chem 136 final projects were performed during the middle of the quarter. Chem 136 had a different emphasis than the Chem 135 predecessor. Chem 136 focused on developing analytical techniques and calculational skills, whereas Chem 135 was more qualitative in nature. Even the projects in Chem 136 emphasized quantitative analysis skills. Though the Chem 136 lab book (86) was used exclusively by Chem 136 students, the level concept was retained to create challenges and motivate the students. The major topic areas covered in Chem 136 were nuclear chemistry, acids and bases, equilibria, kinetics, and electrochemistry. A list of Chem 136 experiments is shown in Table 4 below.

Table 4. Chem 136 Laboratory Schedule.

Week #1, Experiment 1: Some Basic Principles of Nuclear Chemistry (3 levels)

Week #2, Experiment 2: Basic Principles of Colorimetry and Beer's Law (1 level)

Week #3, Experiment 3: Acids and Bases I: Preparation and Standardization
of Solutions (1 level)

Week #4, Experiment 4: Acids and Bases II: Determination of the Percentage of
Acetic Acid in Vinegar (2 levels)

Week #5, Experiment 5: Kinetics and Equilibria of CO_2 Reactions

Week #6: Project

Week #7: Project

Week #8, Experiment 6: Using Colorimetry to Determine an Equilibrium Constant
(3 levels)

Week #9, Experiment 7: Electrochemistry (3 levels)

Every experiment in Table 4 involved the use of the MSU laboratory interface. In every experiment *except* for nuclear chemistry, students had the opportunity to design their own experiment program with the lab interface.

The Final Projects

The final projects were used in Chem 125/135 to bring all of the *conceptual,*
laboratory, and *computer skills* together for the students. For the Chem 125/135 and
Chem 136 students, the final projects also allowed them to participate in the *process of*
science as described in Chapter 4 of this thesis. Students selected a chemical research
problem and were instructed to design an experiment, using the MSU lab interface
system, to fulfill the objectives proposed in the research problem. The Winter quarter
versions of the Chem 125/135 projects are given in Appendix A. The Chem 136 final
project handouts appear in Appendix B. Appendices A and B are the project write-
ups exactly as given to the students. All references and figure numbers in each
project are self-contained. Table 5 is a listing of the Chem 125/135 projects, followed
by a listing of the Chem 136 projects in Table 6.

Table 5. Chem 125/135 Final Projects.

1. Turbidimetric Determination of Sulfate in Water
    (Difficulty Factor: 1.15)

2. Determination of Beta Radiation Penetration (Autumn)
    Thermometric Determination of Chloride Concentration in Water (Winter)
    (Difficulty Factor: 1.10)

3. Determination of Copper(II) in an Unknown
    (Difficulty Factor: 1.05)

4. Determination of Cobalt(II) in an Unknown
    (Difficulty Factor: 1.02)

5. Electrochemistry
    (Difficulty Factor: 1.02)

6. Acid/Base Properties of Over-the-Counter Medicines
    (Difficulty Factor: 1.02)

7. Acid/Base Properties of Common Household Products and Food
    (Difficulty Factor: 1.00)

Table 6. Chem 136 Projects.

1. Kinetics of the Bromine/Formic Acid Reaction
   (Difficulty Factor: 1.05)

2. Thermometric Determination of Chloride Concentration in Water
   (Difficulty Factor: 1.05)

3. Determination of Ammonia in Household Cleaner
   (Difficulty Factor: 1.05)

4. Formation of Complex Ions of Cadmium
   (Difficulty Factor: 1.02)

5. Using a Thermometric Indicator to Determine Acetate Concentrations in
   Unknowns (Difficulty Factor: 1.02)

6. Phosphoric Acid Concentrations in Colas
   (Difficulty Factor: 1.00)

7. Determination of Acid in Toilet Bowl Cleaners
   (Difficulty Factor: 1.00)

The project write-ups give one or two pages of background information, followed by a short explanation of experimental procedures, including lab interface preliminaries. These procedures are purposely vague to give students a chance to develop their own methods for meeting the objectives. Finally, the students are given a list of minimum requirements and references. The objectives are well-defined; the method for achieving the objectives is not. Each project was assigned a *difficulty factor*. The difficulty factor was used to reward students for selecting a project which was more difficult to do from a design or experimental technique standpoint. The students' point grade for the project was multiplied by the difficulty factor to arrive at the student's final grade for the project.

In Chem 125/135, students were given 7 hours (two full lab periods and 1/3 of another) to complete their projects. The more experienced Chem 136 students were given 6 hours (two full lab periods). Figure 20 shows a student working in the laboratory on a titration project. Notice that all of the computer equipment is elevated from the bench top, giving sufficient work space to mix chemicals and set up apparatus. At the same time, the computer, monitor, keyboard, and interface are easily accessable and clearly in view.

FIGURE 20. Student at a Laboratory Station.

For all of the final projects, students were required to submit a formal report following the format on the last page of Appendix B. A set format for reports made it easier to grade the reports for the different projects consistently and fairly. The majority of the students typed these reports, though this was not a requirement. One

formal report was turned in per pair of lab students. Both students received the same grade for the final project. A few students voiced disdain for the fact that they had a lazy lab partner who did little of the work, but got the same grade. However, most students felt that this was a fair method of grading.

In addition to the formal reports, Chem 125/135 students were required to give a short (~5 minutes) oral presentation about their project and results to their peers and the lab instructor on the last day of lab. We viewed the oral presentation not only as an opportunity for students to speak in front of a group of people, but to verbally discuss technical information, as well. The oral presentation was worth 10% of the total project grade; the final project was worth about 1/3 of their total course grade. Because of the number of laboratory concepts we wished to cover, Chem 136 students did not give an oral presentation about their projects.

The final projects clearly channeled the students' time into the thought-provoking aspects of experiment design and data interpretation and away from the tedium of data collection and number crunching (see Figure 6). The projects gave students a chance to explore a chemical problem on their own, with little direction. As can be deduced from the project write-ups in Appendices A and B, many of the projects would not have been possible in a conventional, non-MBL general chemistry laboratory. The projects were *not* easy for the students but, as we'll see in Chapter 7, they overwhelmingly favor the final project concept in the laboratory. Chapter 7 will include a discussion of the students' perceptions of our laboratory program.

To give the reader a better understanding of the significance of the lab interface system from an experimental standpoint, I will discuss a variety of experiment projects designed for and performed with the MSU lab interface in Chapter 6. These experiments provided much of the basis for the project and experiment selection for the general chemistry laboratories.

CHAPTER 6

EXPERIMENTS

The speed, accuracy, and precision advantages afforded the microcomputer-based laboratory opens new doors to experimentation in the chemistry laboratory. In addition, old doors are opened further, allowing for deeper exploration into classic chemistry experiments. During my thesis work, I developed, tested, and/or performed over 90 different experiments with the MSU laboratory interface. Though the emphasis was on chemistry, I also developed experiments for physics, biology, and earth science as part of the teacher workshop program (87). In this chapter, I will discuss a few of these experiments and the results as an illustration of the wide range of utility of the MSU interface system in the laboratory. This chapter will include discussions on *thermometry*, *acid-base titration*, *colorimetry*, and *nuclear chemistry* experiments.

Thermometry

The MSU lab interface system makes *thermometry*, the measurement of temperature, a viable and versatile means of studying chemical reactions in the general chemistry laboratory. In conventional laboratories, thermometers are useful in measuring the temperature of solutions, boiling and melting points, and changes in temperature of several degrees or more. A thermistor, like that shown in Figure 21, attached to the MSU lab interface (see Figure 9) can perform all of the previously mentioned functions of a thermometer (but better!) and many more. With the MSU lab interface hardware and software, the thermistor can measure temperatures to 0.01°C and repeat that measurement several times per second. Since nearly all

chemical reactions consume or release measurable amounts of energy, the thermistor can be used to detect even very small changes in temperature during a chemical reaction. These small temperature changes often yield important information about the type of reaction occurring or about the progress of a reaction.
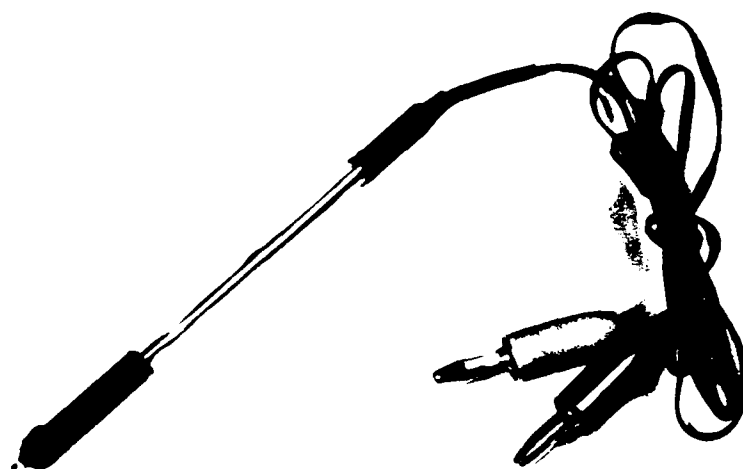
FIGURE 21. A Photograph of a Thermistor.

The use of thermistors for laboratory use has been well characterized by Powers (88) and undoubtedly by many others. The thermistor is simply a *thermal resistor* made of semiconductor ceramic material. The sensing element is the small black bead encased in glass shown at the tip of the thermistor in Figure 21. The MSU lab interface is used to supply a small, constant voltage to the thermistor. Most thermistors, including the ones used in these experiments, have negative temperature coefficients; that is, as the temperature rises, the resistance decreases. There is an exponential relationship between the resistance and temperature, as shown in Figure 22. Likewise, because of Ohm's Law, there is an opposite relationship between current (which the interface actually measures) and temperature.
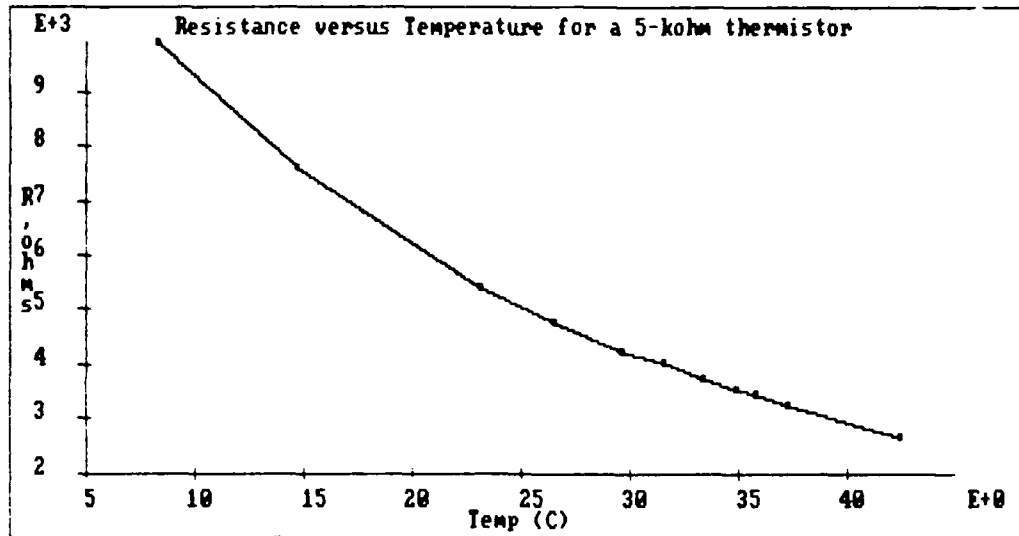
FIGURE 22. Resistance Versus Temperature for a 5-kohm Thermistor.

The mathematical relationship which describes the behavior of thermistors, and is seen in Figure 22 above, is as follows (88):

$$R(T_1)/R(T_2) = \exp[B(1/T_1 - 1/T_2)]$$

where $R(T_1)$ is the resistance in ohms at the Kelvin temperature T_1, $R(T_2)$ is the resistance at the Kelvin temperature T_2, and B is a constant which depends upon the material used to make the thermistor.

Although the response of the thermistor is not linear with temperature, this can easily be compensated for in the software. If we take the natural logarithm of both sides of the above equation, we get the following:

$$\ln[R(T_1)/R(T_2)] = B(1/T_1 - 1/T_2)$$

Using the above equation, we see in Figure 23 that a plot of ln(R) versus 1/T

yields a straight line with a *slope of B*. The beta value *B* for the *5-kiloohm thermistor*

shown in Figure 21 and tested in Figures 22 and 23 is 3393 K. Higher resistance

thermistors have larger *B* values and higher temperature operating ranges.
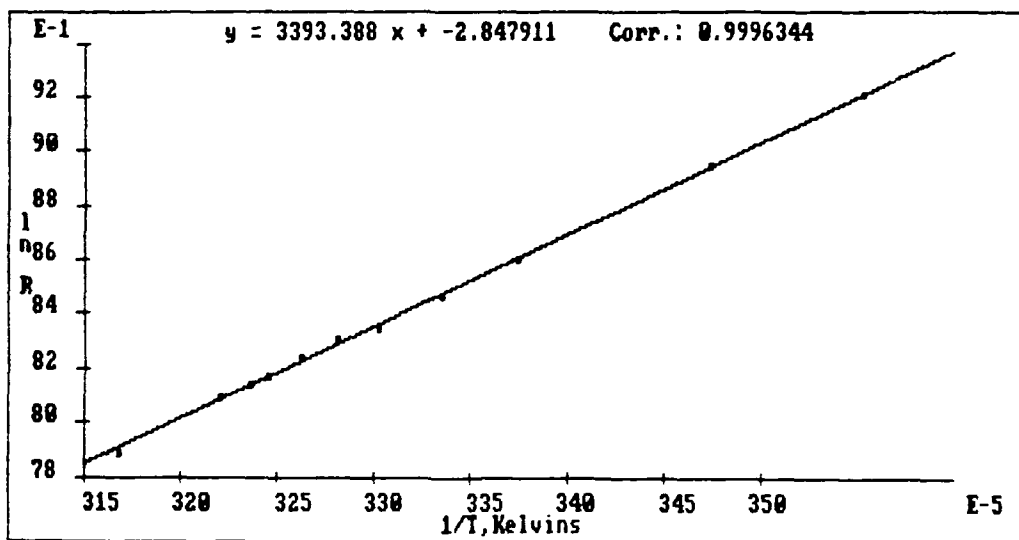
FIGURE 23. Ln(R)   sus 1/T for a 5-kohm Thermistor.

The thermistor can be easily calibrated by measuring the resistance (or current

at a constant voltage) and finding a value for *B*. Once this calibration constant is

calculated, the MSU lab interface software can convert current readings from the

thermistor into corresponding temperatures. It should be pointed out that linear

response solid-state temperature sensors (such as the Analog Devices LM335) do exist

and have also been interfaced with personal computers (89).

The data obtained in Figures 22 and 23 are quite easy to obtain with the MSU

lab interface program. Teachers have obtained the operating parameters of their

thermistors during our workshops described earlier. Figure 24 is the experiment

program listing which obtained this data. The key lines are Line 2, which sends a

voltage (1000 mV) to the thermistor; Line 7, which sends the voltage input to Column

A or a **Spreadsheet** data file; Line 8, which sends the current (I2) value to Column B of a data file; and Line 9, which sends a temperature to Column C. The remaining lines are mostly lines used to make the program friendly and to provide on-screen data to the user.

```
1 CLEAR TEXT SCREEN
2 SEND 1000.000000 TO DAC2
3 PRINT "Press the "w" button to send a voltage and current" ON LINE 1
4 PRINT "from DAC2 and I2 to Col A & B." ON LINE 2
5 PRINT "The temperature (C) will be sent to Col-C" ON LINE 4
6 WAIT UNTIL INPUT FROM SwW = Pressed
7 SEND INPUT FROM DAC2 TO COL-A
8 SEND INPUT FROM I2 TO COL-B
9 SEND INPUT FROM Temp1 TO COL-C
10 PRINT "DAC2" ON LINE 10
11 PRINT INPUT FROM COL-A ON LINE 11
12 P  NT "I2" ON LINE 13
13    INT INPUT FROM COL-B ON LINE 14
14 PRINT "Temperature (C)" ON LINE 16
15 PRINT INPUT FROM COL-C ON LINE 17
16 PRINT "Flip up the x-switch to stop taking data." ON LINE 21
17 IF INPUT FROM SwX = On GOTO 19
18 GOTO 6
19 STOP
```

FIGURE 24. Experiment Program to Obtain Thermistor Operating Characteristics.

Because of the resolution of the thermistor and the sensitivity to small temperature changes, students can study chemical reactions in a completely different light than in a conventional laboratory. An excellent example of this is in the study of the reaction of silver ion (Ag^+) with chloride ion (Cl^-).

The Ag^+/Cl^- Reaction

The reaction between silver ion and chloride ion is commonly studied in the general chemistry laboratory. Silver chloride (AgCl) has a K_{sp} of 1.8×10^{-10}, making the formation of the white AgCl precipitate a common test for the presence of silver in qualitative analysis.

Because of the extreme insolubility of silver chloride, gravimetric analysis using the Ag^+/Cl^- reaction is also popular. Kennedy et.al. detail a gravimetric analysis experiment using pure silver to determine the empirical formula of silver chloride (90). The experiment requires the use of at least 0.5 g of silver and the formation of approximately 0.7 g of AgCl for an effective experiment. In addition, obtaining the silver chloride precipitate for weighing requires very good filtration techniques.

Chloride ion concentration can also be found by gravimetric analysis by precipitation as silver chloride with the addition of a solution of silver nitrate to the chloride-containing solution (91). Chloride ion is one of the major inorganic ions found in water and wastewater. In drinking water, the salty taste produced by chloride concentrations is variable and depends on the chemical contents of the water. Some water samples containing 250 mg chloride/liter may have a detectable salty taste if the cation is sodium. However, if the cations are calcium and magnesium, the salty taste may be absent in concentrations up to 1000 mg chloride/liter (92). High chloride content in water can be detrimental to metallic pipes and structural material, as well as plants. Titrimetric analysis for chloride is more common than gravimetric. Several titrimetric methods involve the determination of a color endpoint (92). These methods require considerable solution preparation and considerable practice. The methods are quite accurate (better than 10 mg/L) in the 100-1000 mg chloride/L range.

We determined that if the chloride ion concentration is higher than 1000 mg/L, a much easier and faster method of determining chloride ion concentration is by a *thermometric* titration. Quite simply, the endpoint of the titration is determined by examining the changing slope of the temperature versus volume of titrant curve. The method requires one-fifth the amount of silver required for a gravimetric analysis and gives similar results.

To determine the chloride ion concentration thermometrically, the chloride-containing sample is titrated with a known concentration of silver nitrate ($AgNO_3$) solution. The following reaction occurs:

$$AgNO_3(aq) + Cl^-(aq) \text{-----}> AgCl(s) + NO_3^-(aq)$$
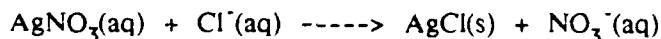
As the above reaction proceeds, a white precipitate of AgCl forms. In addition, the reaction is exothermic (ΔH° = -65.5 kJ), so heat is evolved. As heat is evolved, the temperature of the reaction mixture will rise. When the reaction is complete (all of the chloride has been converted to AgCl), heat is no longer evolved and the temperature of the mixture stabilizes or begins to drop. The point at which the temperature stabilizes is the endpoint. With our students, small volumes (20 mL) of solutions at low chloride concentrations (0.04 - 0.12 M) are used, resulting in small (usually < 1°C) temperature changes. However, these small changes are ideally suited for measurement by a thermistor and the lab interface.

An example of the Ag^+/Cl^- thermometric titration is shown in Figure 25. In this example, the known sodium chloride solution (0.0500 M) was added to 20.0 mL of the "unknown" $AgNO_3$ solution (~0.05 M). The experiment was performed in a double-nested styrofoam cup, with the thermistor resting in a stationary position in the cup. The solution was hand-stirred during the titration with a glass stirring rod. A constant flow buret was set up by measuring the flow rate three times between two set marks on a normal buret. The volume of the buret was then kept between these two marks during the titration, giving a constant head pressure. The temperature was measure approximately every 0.8 seconds (which corresponded to 0.1 mL of titrant). During the titration, 374 time/temperature data points were obtained and were converted to volume/temperature data points using the flow rate and the **Spreadsheet**.

As seen in Figure 25, the total temperature change during the titration was only about 0.8°C, much too small to be measured accurately with a standard mercury thermometer. The thermometric endpoint is shown with the black arrow. The results of the titration were quite repeatable, giving a concentration of 0.0563 +/- 0.0012 M for the "unknown" $AgNO_3$ solution.
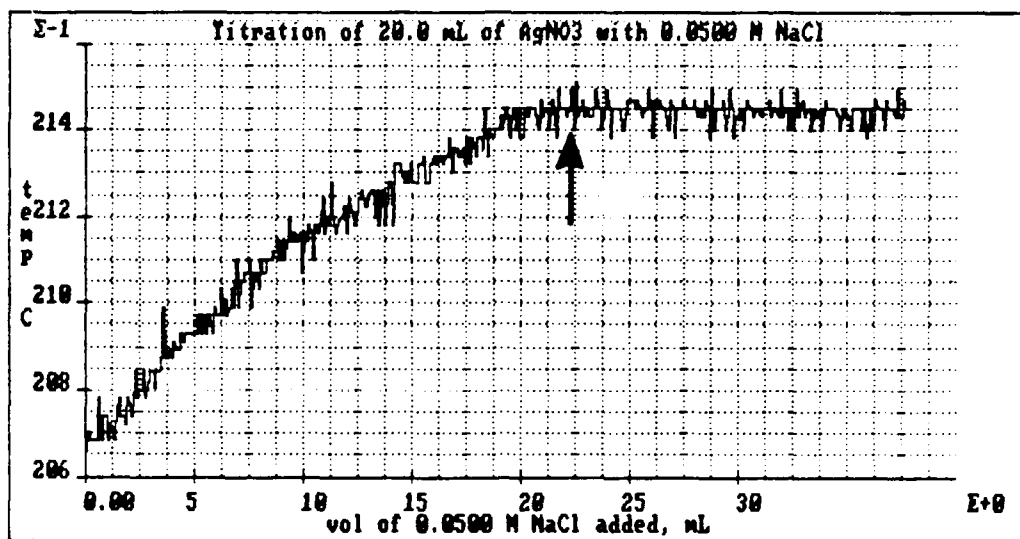
FIGURE 25. Ag⁺/Cl⁻ Thermometric Titration.
The arrow indicates the endpoint of the reaction.

Figure 26 shows the experiment program which obtained the data in Figure 25. Note that this simple program is very similar to ones which students see as examples in their laboratory computer book, as discussed in Chapter 5.

```
1 CLEAR TEXT SCREEN
2 WAIT UNTIL INPUT FROM SwW = Pressed
3 START Timer1 FOR sec
4 PRINT INPUT FROM Temp1 LARGE ON LINE 1
5 SEND INPUT FROM Timer1 TO COL-A
6 SEND INPUT FROM Temp1 TO COL-C
7 DELAY 250 millisec
8 IF INPUT FROM SwX = On GOTO 10
9 GOTO 4
10 STOP
```

FIGURE 26. Experiment Program for Thermometric Titrations.

Figure 27 shows the appearance of the **Spreadsheet** when converting the time data (in Column A) to volume data (in Column D) using the **Analysis** option. A box appears for the student to type in the formula he/she desires. Note that Column D

will be equal to whatever formula is typed into the box. Information in square

brackets are comments which are not used in the actual calculation. The actual

formula is a*2.00/18.49, which is multiplying all of the time values in Column A by

the flow rate of the buret (2.00 mL every 18.49 seconds). The lower box shows the

functions and operators available for students to use in their formulas.

```
Column Setup  Acquisition  Analysis  Graph  Stats  Print  File  Quit
```

Data Acquisition				Data Analysis	
No.	A: time (se	B	C: temperat	D: [vol of	E	F
347	279.4722		21.2200	30.2295		

```
Enter formula for column D:
D = [vol of NaCl added, mL]a*2.00[ml]/18.49[sec]
```

```
Functions, operators, constants, and column letters may be used in formulas.

Functions available are:
    ABS         COS      DERIV    FACT      LN     SIN    SQRT    STEPU
    ARCTAN      DELTA    EXP      INTEG     LOG    SQR    STEPD   SUM

Operators available are:
    (+)   addition       (-)         subtraction      (*)    multiplication
    (/)   division       (^ or **)   exponentiation   (DIV)  integer division
    (MOD) remainder only
```

```
1       2       3        4 Grid  5Graph   6D Row  7I Row  8       9 Help 10 Quit
```

FIGURE 27. Appearance of the **Spreadsheet** in the **Analysis** Mode.

As shown by this example, thermometry is a powerful tool for quantitative

analysis, being as accurate and much simpler than gravimetric and classical titrimetric

methods. This particular reaction was adapted as a final project for Chem 125/135

and Chem 136. As we will see later in the section on acid/base titrations,

thermometry can also be used to complement a potentiometric titration.

Formation of Complex Ions of Cadmium

Thermometry can also be used to monitor the formation of complex ions. In

the case of the formation of complex ions between cadmium (Cd^{2+}) and iodide (I^-), all

of the possible complex ions are colorless. Therefore, there are no color indications of

complex ion formation in the reaction of cadmium with iodide. In addition, none of

the species formed (i.e. CdI_2) are precipitates. Moving boundary, conductivity, and spectral studies have been used to determine which complex ions are actually formed, but are certainly beyond the ability of introductory chemistry students (93,94). By examining the thermodynamics of the complex ion formation, thermometry can play a role in determining the species which are produced. Although thermometric analysis of the Cd^{2+}/I^- system is less versatile than conductance and spectral studies, it is also much simpler.

Studies by Alberty and King (93) and others (94,95) indicate that the species CdI^+, CdI_2, CdI_3^-, and CdI_4^{2-} are all present in solutions of cadmium and iodide, with Cd^{2+} and I^- also being present in dilute solutions. The greatest amount of iodide complex in solution appears to be CdI_4^{2-} (95). Hill and Magee (96) state that there is also evidence for the CdI_5^{3-} specie, but I could find no other evidence for the presence of this specie in the literature. Aylett notes, in a comprehensive review of cadmium halide complexes, that "species with more than four halogens per cadmium do not seem to exist in aqueous solution" (94).

The equilibrium constant data in Table 7 provides useful information about the species in solution. The constants given are cumulative, or *gross constants*, B_n, where

$$B_n = [CdI_n]^{2-n}/[Cd^{2+}][I^-]^n$$

for the reaction $Cd^{2+} + nI^- \rightleftharpoons CdI_n^{2-n}$.

Table 7. Equilibrium Constant Data for Cadmium Iodide Complex Ions.

Ref.	Ionic Strength	B_1	B_2	B_3	B_4
93	~1	83	1600	2.6×10^4	1.20×10^6
94	1	81	2200	4.5×10^4	7.2×10^5
94	2	100	5700	1.0×10^5	6.5×10^5

As shown in Table 7, the formation of all of the species are favored, from CdI^+ to CdI_4^{2-}. The complex ions also have associated with them a *heat of formation*. If the formation of the products are thermodynamically favorable, heat is released, causing a temperature rise in the solution. Table 8 is a summary of some thermodynamic values for cadmium iodide complex ions (94). Note especially the ΔH_f° value associated with CdI_4^{2-}.

Table 8. Thermodynamic Values for Cadmium Iodide Complex Ions.

	ΔH_f° (kJ mol^{-1})	ΔG_f° (kJ mol^{-1})	S° (J K^{-1} mol^{-1})
CdI^+ (aq, M = 1)	-141	-141	43.1
CdI_3^- (aq, M = 1)	---	-259	---
CdI_4^{2-} (aq, M = 1)	-342	-316	326

As long as *sufficient iodide is provided to a solution* of cadmium ions, heat will be released and stable products will form. When no new complex ion species are formed, the solution mixture should cease to release heat and the temperature of the solution should stabilize or go down. It is at this point that a formula for a thermodynamically stable complex ion can be determined based on the amount of available cadmium cation and the amount of added iodide anion. As indicated in the literature, the predominate specie is CdI_4^{2-}; thus, a temperature curve for the addition of I^- to Cd^{2+} should give a "break" or leveling at four stoichiometric equivalents of I^-.

In this experiment, I prepared a 1.00 M solution of $Cd(NO_3)_2 \cdot 4H_2O$ and a 2.01 M solution of NaI. (Early attempts at this experiment using 0.1 M Cd^{2+} and 0.2 M I^- were unsuccessful; the temperature change during the reaction was too small.) I placed 10.0 mL of the 1 M Cd^{2+} solution into a double-nested styrofoam cup and rested the thermistor on the bottom of the cup. Based on the 4:1 I^-/Cd^{2+} stoichiometry

indicated in the literature, a temperature curve "break" should occur after

approximately 20.0 mL of I⁻ solution has been added to the 10.0 mL of Cd^{2+}. The

experiment, then, was performed in an identical manner to that of the Ag^+/Cl^-

experiment described in the previous section. Figure 28 shows the results from a

timed titration (times being converted to volumes via the **Spreadsheet**, as in Figure 27)

in which 302 data points were obtained in 239 seconds. The experiment program was

identical to that in Figure 26.

FIGURE 28. Determination of Cd^{2+}/I^- Complex Ion Formation.
The arrow indicates the point at which four equivalents of I⁻ have been added.

The black arrow in Figure 28 indicates on the curve where the 4:1

stoichiometric equivalence point would be for the formation of CdI_4^{2-}. It is clear at

this point that the temperature of the reaction mixture has leveled out, and begins to

tail down after the addition of about 2.5 mL additional NaI. The tailing down is due

to the fact that the NaI solution being added to the reaction mixture *after* the

stoichiometric equivalence point was *cooler* than the reacting mixture (the Cd^{2+} and

NaI solutions started out at the same temperature). Since CdI_4^{2-} is the highest complex

ion formed, no further reactions take place with the addition of more NaI. So, the addition of the cooler NaI does *not* generate more heat; it cools the reaction mixture down.

As seen in Figure 28, the temperature of the reaction mixture changes by nearly $3°C$. With this "large" of a temperature change, it seemed viable to attempt a manual titration; that is, add NaI with a buret in volume increments rather than constant flow, entering the total volume added via the keyboard after each increment. Although this method requires about three times the time required for a timed titration, there should be sufficient heat released to see the thermometric endpoint. Figure 29 shows a manual titration during which 40 data points were obtained. The black arrow shows the stoichiometric endpoint for the formation of CdI_4^{2-}. Note that the curve is nearly identical to Figure 28.

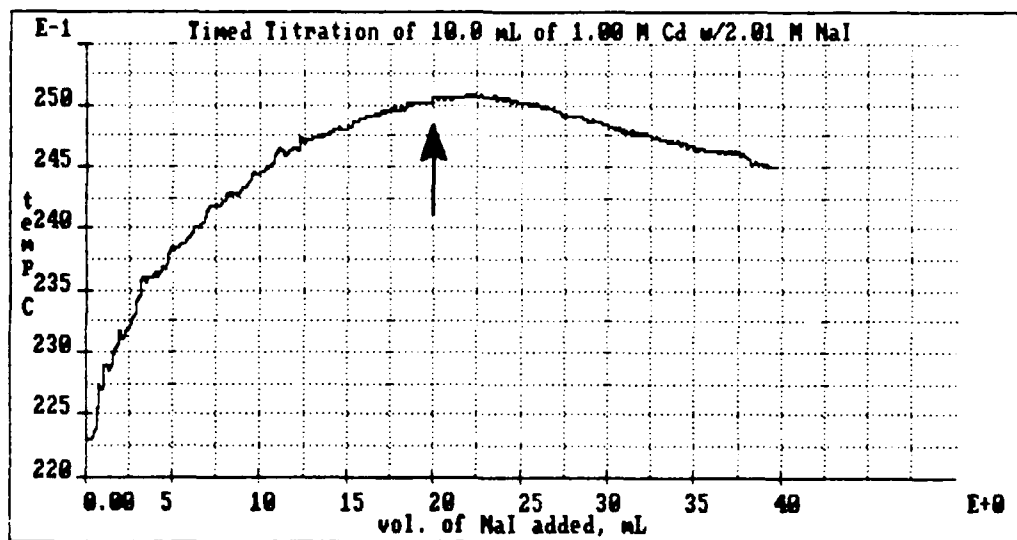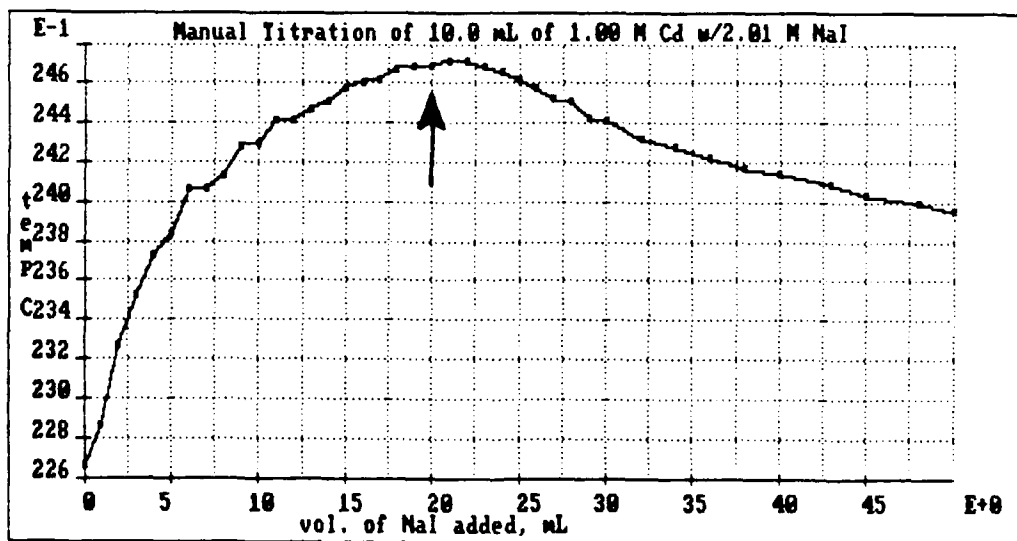**FIGURE 29.** Manual Titration to Determine Cd^{2+}/I^- Complex Ion Formation. *The arrow indicates the point at which four equivalents of I^- have been added.*

Based on the data in Figures 28 and 29, a clear case can be made for CdI_4^{2-} as being the major complex ion formed in the reaction between Cd^{2+} and I^-. This experiment was adapted as a project for Chem 136 (see Appendix B). This is *not* an

easy project for students. Most students are not clear with respect to what they "should" be observing; it is truly an experiment for the students. In addition, the starting temperature of the reactants will affect the shape of the curve, as will mixing. This is an ideal experiment for using the lab interface and incorporating the process of science. There are several ways to *design the experiment* to get the data you need to find the formula for the most stable complex ion. Excellent data can be *gathered* quickly via a timed titration, or more slowly with a manual titration. In the most thought-intensive aspect, the temperature data can be quickly graphed to *analyze the data* to determine the correct complex ion formula. If the data is inconclusive, the experiment can quickly be repeated and the new data graphed and analyzed.

The MSU lab interface makes thermometry a tremendous tool to study chemical reactions at the general chemistry level. Students find out that the small temperature changes being produced during reactions *are* meaningful! As we will see in the next section, thermometry also provides complementary information to pH data in an acid/base titration. Fast, accurate measurements of temperature yield information previously unavailable to beginning chemistry students. As Lord Kelvin stated in 1871,

> Accurate and minute measurement seems to the non-scientific imagination a less lofty and dignified work than looking for something new. But nearly all the grandest discoveries of science have been but the rewards of accurate measurement and patient long-continued labor in the minute sifting of numerical results (90).

The computer is providing the accuracy in the measurement *and* the patience for sifting through and manipulating numerical results.

Acid/Base Titrations

Acid/base titrations are among the most commonly performed experiments in general chemistry. Many important chemical concepts can be learned from acid/base titrations, including stoichiometry, balancing equations, equilibrium, and acid/base

reactions. In almost every acid/base titration done in a conventional laboratory, the equivalence point is estimated by determining the indicator color endpoint of the titration. While this is an important and extremely useful technique, much of the chemistry of the reaction is lost. A potentiometric, or pH, titration yields much more information about the chemical reaction. However, a good pH titration requires a steady hand and patience, neither of which most students possess. The successful pH titration curve is a "work of art", requiring at least 30 data points and at least that many minutes just to obtain that one set of data points. In addition, the pH titration curve can take a considerable time to graph by hand. While taking the data from a pH meter and graphing it by hand is *not* inherently bad, it does consume a considerable amount of time which could be spent analyzing the data. The lab interface is again the ideal tool to shift the students' time from the tedious tasks to the thought-provoking ones.

The Strong Acid/Strong Base Titration

Perhaps the most commonly observed titration in introductory chemistry is that of a strong acid with a strong base. The most commonly used strong acid is hydrochloric acid (HCl) and the most commonly used strong base is sodium hydroxide, (NaOH), which react as follows:

$$HCl(aq) + NaOH(aq) \longrightarrow NaCl(aq) + H_2O(l)$$
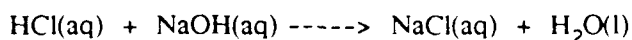
The above reaction is so commonly used to demonstrate titration techniques because the reactants combine on a one-to-one molar ratio and the pH of the solution is 7 when the moles of base added are equal to the moles of acid in the starting solution (the *equivalence point*). Students, usually from past experience, associate solutions of pH 7 with being neutral, which fits with the definition of the acid/base reaction being a neutralization reaction. Of course, all acid/base reactions do *not* give a pH 7

solution at the equivalence point; only those between strong acids and strong bases. This reaction was chosen as an example for this thesis primarily to show the data acquisition and analysis capabilities of the MSU lab interface system.

Figure 30 shows the experimental set-up used to obtain the titration data presented in this section. Note that the apparatus is "designed" as simply as possible. The double-nested styrofoam cup makes it possible to obtain meaningful temperature information. Stirring is crucial in such experiments; hand-stirring with a glass stirring rod seems to work fine.
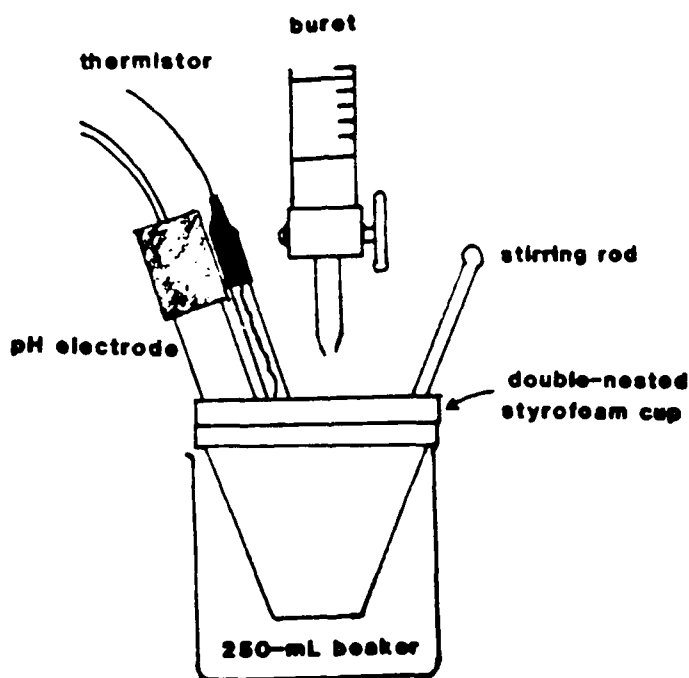
FIGURE 30. Experimental Set-up for Acid/Base Titrations.

In this strong acid/strong base example, 30.0 mL of approximately 0.1 M HCl was titrated with standardized 0.101 M NaOH. A timed titration was performed, with 425 data points obtained in 342 seconds. The times were later converted to volumes in the **Spreadsheet** using the flow rate of the buret. Figure 31 is a pH versus volume of titrant plot for this titration.
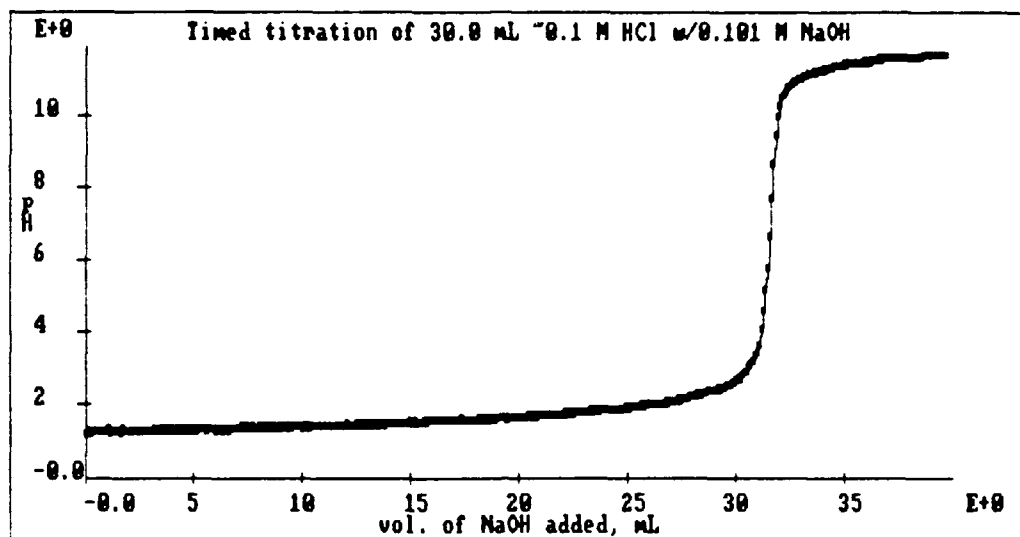
FIGURE 31. pH Versus Volume for a HCl/NaOH Titration.

It should be noted again that 425 data points were obtained in under six minutes. In a conventional potentiometric titration using a pH meter, one would be lucky to get 30 data points in a half-an-hour. This titration can be done manually with the lab interface system by entering volume data point-by-point. Because the pH (and temperature) information is still obtained via the system, 50 data points can be obtained in about 15 minutes with very good results. Notice in Figure 31 that the system obtained 9 data points on the steep part of the curve, in the vicinity of the equivalence point. In a manual titration, these are the most time-consuming and difficult points to obtain; it is not unusual to obtain 5 or less points in this region manually.

As mentioned previously, the equivalence point of a strong acid/strong base titration curve occurs at a pH of 7. For titration curves in general, the equivalence point is the steepest part of the curve. In Figure 31, it is relatively simple to "eyeball" the steepest part of the curve. However, the **Spreadsheet** allows students to find the steepest point mathematically using a derivative function. Figure 32 is a plot of the

same pH/volume curve as in Figure 31, with the derivative of the curve superimposed. The black arrow points to where the derivative function has its highest value.
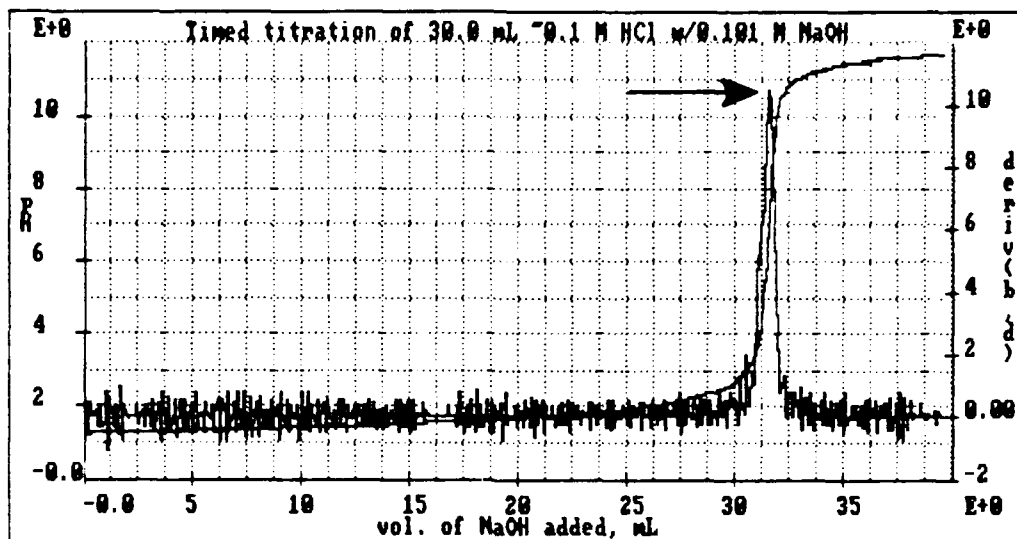
FIGURE 32. pH Versus Volume Curve for HCl/NaOH with Derivative. The arrow shows the maximum point on the derivative curve.

The key value in the titration is the volume of 0.101 M NaOH added which corresponds to the steepest part of the titration curve. From Figure 32 (and the raw tabular data), this corresponds to a volume of 31.49 mL, giving the HCl solution a molarity of 0.106 M.

As shown in Figure 30, temperature data was also obtained during the titration. Acid/base reactions are exothermic, meaning the solution temperature will rise as titrant is added, but level out or decrease after the reaction is complete at the equivalence point. In other words, the pH and temperature data should be complementary. Figure 33 shows this to be the case. Notice that the temperature rise in Figure 33 is not linear. The rise flattens out as the total volume of the solution increases. Essentially, the heat released during the reaction must heat up a greater volume of solution as the titration proceeds. The downward turn of the temperature curve is very evident around the equivalence point. This downward turn occurs

because the reaction between HCl and NaOH is complete. The reaction mixture is cooling off due to heat loss to the environment and/or due to the fact that the additional NaOH being added is slightly cooler than the reaction mixture. Figure 34 illustrates the location of this downward turn even better.
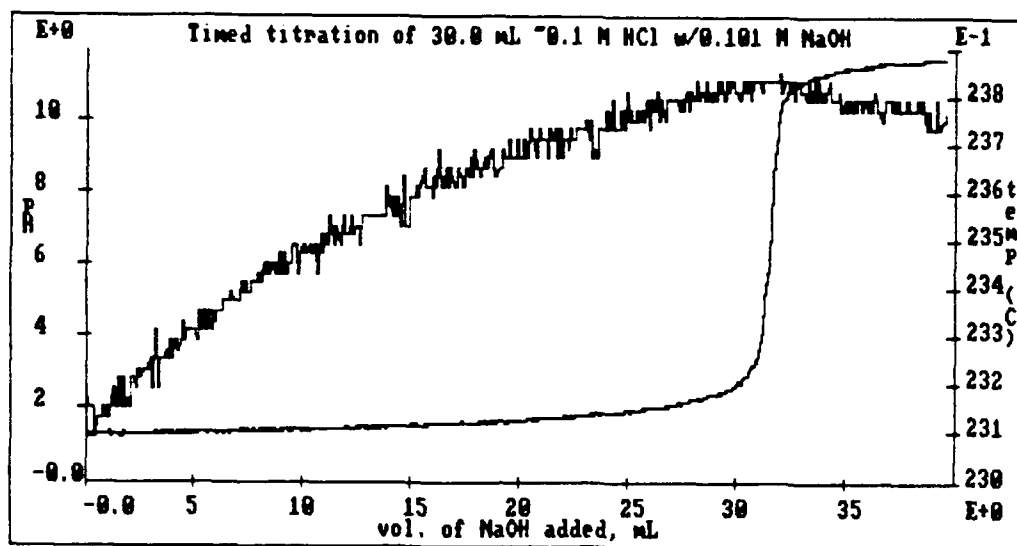
FIGURE 33. Temperature and pH versus Volume for the HCl/NaOH Titration.
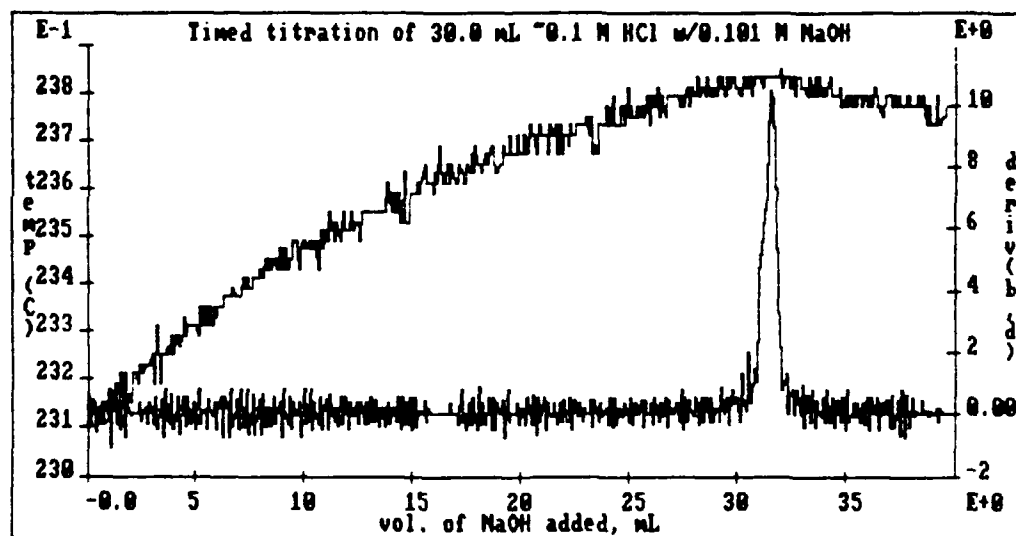
FIGURE 34. Temperature and Derivative Curves Superimposed.

The derivative of the pH versus volume curve literally points to the location of the change in the temperature curve in Figure 34. As you can see by this example, supported by the graphs in Figures 31-34, the MSU lab interface package provides a fast, accurate, and novel way to explore acid/base reactions. The experiment can be *designed* to take data at the desired rate. The data can be *acquired* via a **Spreadsheet** data file and displayed on the monitor as the reaction is occurring. As illustrated in Figures 31-34, the data can then be *analyzed* in detail with the **Spreadsheet**, yielding information normally not obtainable during an acid/base titration or, at the very least, conventionally difficult to obtain. This titration technique for strong acids and strong bases was used as an exercise in experiment design during our second round of teacher workshops, as well as a final project in Chem 125/135 and Chem 136 (see Appendices A and B).

Weak Acids and Bases

The titration techniques using the MSU lab interface system work equally well with weak acids and bases. In addition to finding unknown concentrations using the equivalence point, the pH curves are also useful in determine the acid dissociation constants (K_a) for weak acids. In the remainder of this section, I'll present titration results from some representative weak acid/base systems.

Acetic Acid (Vinegar). Figures 35 and 36 show the results of a timed titration of 8.640 g of white vinegar diluted with 20 mL of water, then titrated with 0.300 M NaOH. The reaction occurring is as follows:

$$CH_3COOH(aq) + NaOH(aq) \longrightarrow CH_3COONa(aq) + H_2O(l)$$
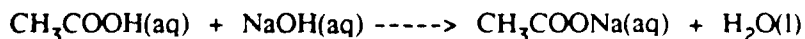
The temperature endpoint is evident in Figure 35, but is strikingly clear when superimposed on the pH/volume derivative curve in Figure 36.
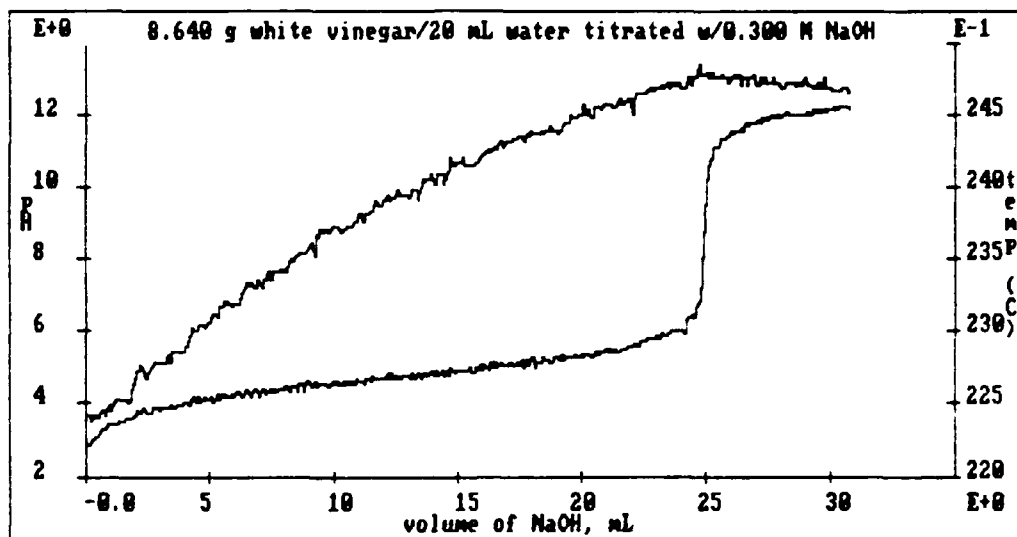
FIGURE 35. pH and Temperature Curve for Vinegar Titration.
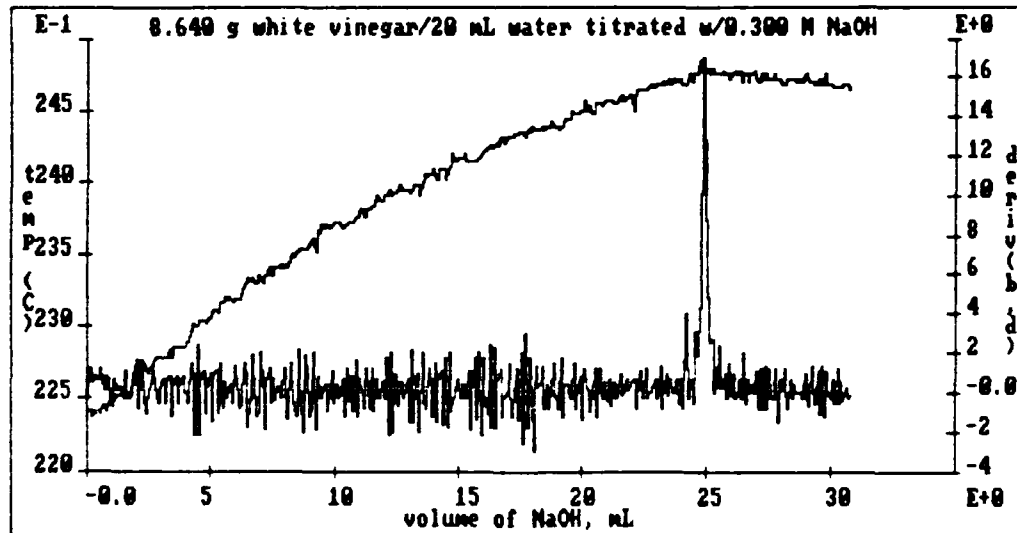
FIGURE 36. Temperature and Derivative Curve for Vinegar Titration.

Using the above graphs, the percentage of acetic acid in the vinegar sample was determined to be 5.19%, reproducible to +/- 0.05%. According to the bottle, the

vinegar is at least 5% acetic acid by weight. The pK_a determined from the half-equivalence point was 4.66. The actual pK_a for acetic acid is 4.74, equivalent to a K_a of 1.8 X 10^{-5} (97). As seen below, using a 2.566 g sample of the same white vinegar diluted with 28 mL of water and titrated with 0.101 M NaOH, a manual titration with 31 data points still gives excellent results. Although much rougher in the manual titration, the temperature endpoint is still quite clear in Figure 37. From this data, the percentage of acetic acid was determined to be 5.18%. The pK_a from the half-equivalence point was 4.63. This particular titration was adopted as an experiment for Chem 136 during the Winter 1990 quarter.
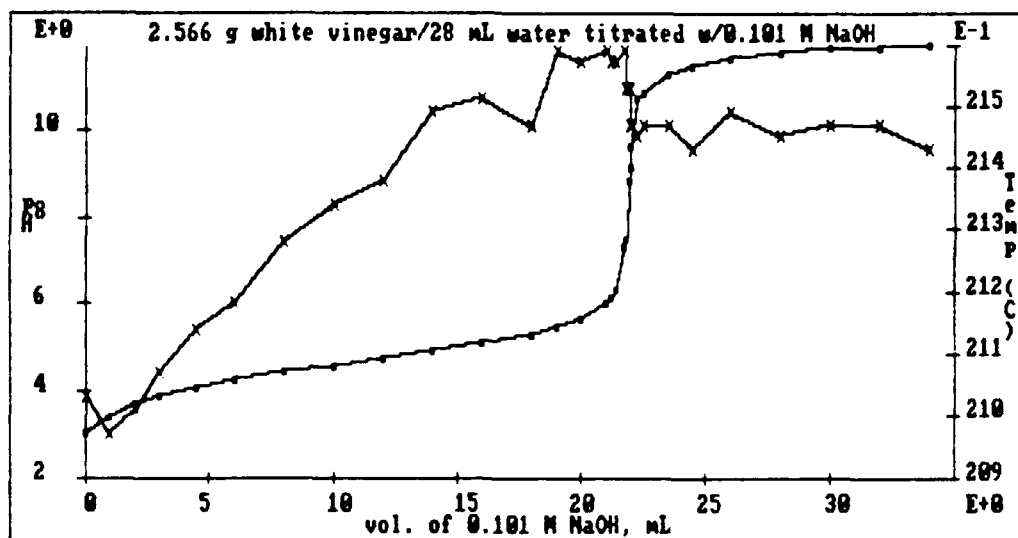
FIGURE 37. Temperature and pH Curves for a Manual Vinegar Titration.

Phosphoric Acid. A polyprotic weak acid gives equally good results. As with all polyprotic acids, phosphoric acid releases its protons step-wise as shown below (97):

$$H_3PO_4(aq) + H_2O(l) \rightleftharpoons H_3O^+(aq) + H_2PO_4^-(aq)$$
$$K_{a1} = 7.5 \text{ X } 10^{-3}$$
$$pK_{a1} = 2.12$$

$$H_2PO_4^-(aq) + H_2O(l) \rightleftharpoons H_3O^+(aq) + HPO_4^{2-}(aq)$$
$$K_{a2} = 6.2 \text{ X } 10^{-8}$$
$$pK_{a2} = 7.21$$
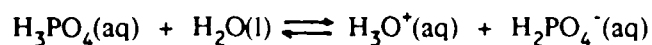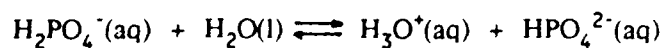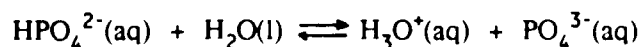
$$HPO_4^{2-}(aq) + H_2O(l) \rightleftharpoons H_3O^+(aq) + PO_4^{3-}(aq)$$
$$K_{a3} = 3.6 \text{ X } 10^{-13}$$
$$pK_{a3} = 12.44$$

The reactions of the base with the protons, then, are as follows:

$$H_3PO_4(aq) + OH^-(aq) \dashrightarrow H_2O(l) + H_2PO_4^-(aq)$$

$$H_2PO_4^-(aq) + OH^-(aq) \dashrightarrow H_2O(l) + HPO_4^{2-}(aq)$$

$$HPO_4^{2-}(aq) + OH^-(aq) \dashrightarrow H_2O(l) + PO_4^{3-}(aq)$$
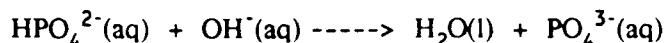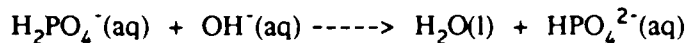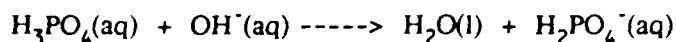
Theoretically, then, one should be able to observe *three* equivalence points for

phosphoric acid when it is titrated with a strong base; one point for each equivalent of

protons consumed by the base. In reality, only two equivalence points are normally

observed since pK_{a3} is so large (the large pK_{a3} of 12.44 means that the concentration

of the titrant would have to be at least 1 M NaOH, which would obscure the other two

equivalence points if the concentration of phosphoric acid was 0.1 M).

Figure 38 shows the timed titration of 30.0 mL of 0.1 M phosphoric acid with

0.300 M NaOH. A temperature endpoint for the second equivalence point is clearly

visible at about 30 mL of titrant added. The bowing of the temperature curve is again

due to the fact that the volume of the solution is steadily increasing, resulting in

smaller temperature increases as the reaction proceeds.
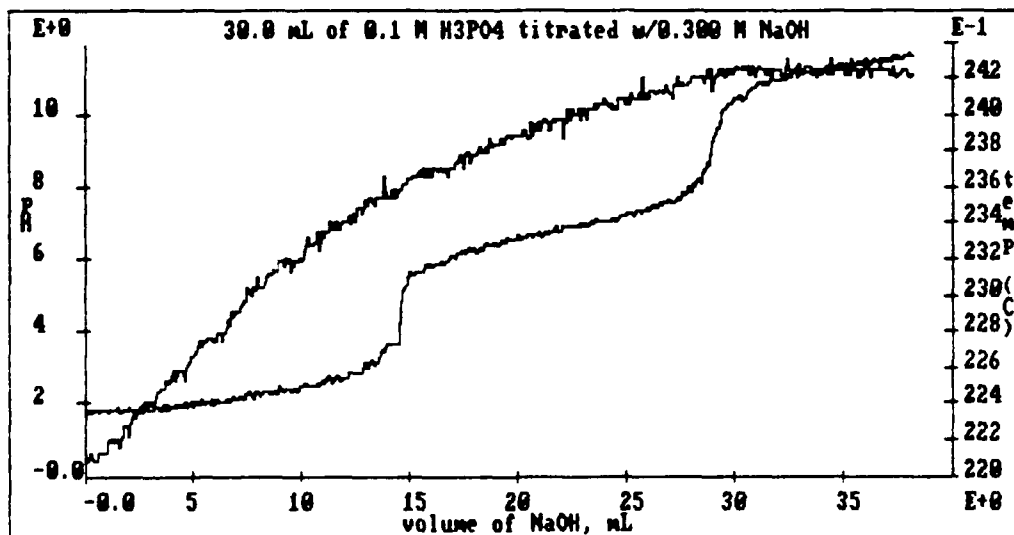
FIGURE 38. Phosphoric Acid pH/Temperature Titration Curves.

From Figure 38 and the tabular data, the first pK_a was determined to be 2.12

(actual, 2.12) and the second pK_a was found to be 6.79 (actual, 7.21). Such a titration

curve clearly demonstrates the fact that phosphoric acid is a polyprotic acid to

students. The phosphoric acid titration was incorporated into Chem 136 as a higher-

level of an acid-base experiment. In addition, the same titration method was used as a

Chem 136 final project to determine the concentration of phosphoric acid in cola

products (see Appendix B). The student results from the cola titrations were clearly

superior to those obtained in a conventional manner by Murphy (98).

Carbonate Titration. The titration of a solution of sodium carbonate (Na_2CO_3)

provides an opportunity to observe the reaction between a strong acid (HCl) being

added to a weak base. The following reactions occur during the titration:

$$CO_3^{2-}(aq) + H_3O^+(aq) \rightleftharpoons HCO_3^-(aq) + H_2O(l)$$

$$HCO_3^-(aq) + H_3O^+(aq) \rightleftharpoons H_2CO_3(aq) + H_2O(l)$$
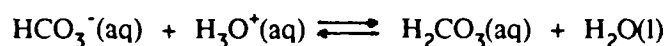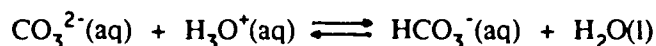
Note that the reverse of these two reactions are dissociation reactions for weak acids.

The pK_{a1} for carbonic acid (the reverse of the second reaction) is 6.37, while pK_{a2}

(the reverse of the first reaction) is 10.32 (97). Figure 39 shows the timed titration

curve for 30.0 mL of 0.1 M Na_2CO_3 with 0.300 M HCl.
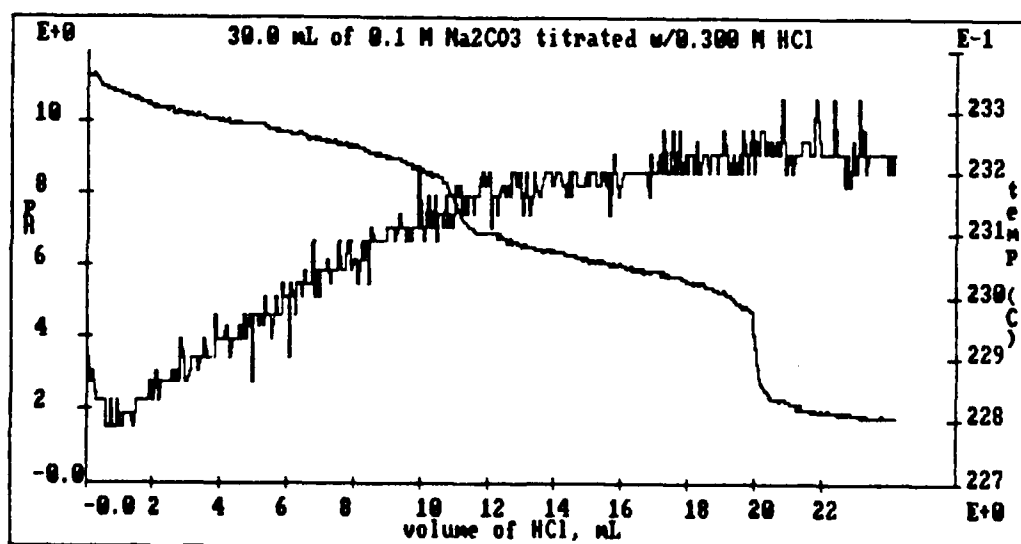
FIGURE 39. Sodium Carbonate pH/Temperature Titration Curves.

The temperature curve in Figure 39 is less definitive than in the previous
titration curves. However, a case can certainly be made for observing a *slight*
downward turn in temperature at the second equivalence point. Using this titration
curve and the tabular data, the first pK_a was determined to be 6.08 (at 15.53 mL), the
actual being 6.37. The second pK_a was determined to be 9.78 (at 5.51 mL); the actual
is 10.32.

Figures 40 and 41 on the following two pages show the MSU lab interface
experiment programs used to obtain the previous acid-base titration data. Figure 40 is
the program for a timed titration, using a constant flow buret. Notice that lines 20-28
are the only real "working" lines. The other 23 lines of the program are user-friendly
lines, including instructions to tell the user what the program does and how to use it.
In Figure 41, lines 19-27 are the working lines. Time is not a variable; the **kbd**
function (line 19) allows the user to input a volume reading from the buret. Although
these programs look more complicated than the ones shown in Chapter 5, they are
really basically the same and were designed in the same manner. These programs
simply contain more user messages.

In these previous acid/base titration examples, I have shown that it is possible
to obtain extremely good results and textbook titration curves using the MSU lab
interface and very simple experimental apparatus. Indeed, many students obtained
similar (if not better!) results employing these techniques in laboratory experiments
and final projects. There is certainly room for improvement; a better means of
obtaining a constant flow buret would be helpful. References 99, 100, and 101 are
just three examples of different methods of setting up an automatic titrator. In
addition, we are actively pursuing the use of a photogate (infrared
transmitter/detector) as a drop counter, since the drop size from a buret tip is very
reproducible from a volume standpoint. The versatility, speed, accuracy, and analysis
capability of the MSU lab interface system turns "simple" acid/base titrations

```
1 CLEAR TEXT SCREEN
2 PRINT "This program is used to measure pH and temp" ON LINE 1
3 PRINT "versus time for an acid/base titration." ON LINE 2
4 PRINT "Before starting the experiment, obtain a" ON LINE 4
5 PRINT "constant flow rate with your buret into" ON LINE 5
6 PRINT "a separate beaker, at 2 to 5 drops/sec." ON LINE 6
7 PRINT "Once you've established a flow rate," ON LINE 9
8 PRINT "swing the buret to the flask which" ON LINE 10
9 PRINT "contains your unknown acid or base." ON LINE 11
10 PRINT "Press the "W" button on the interface" ON LINE 14
11 PRINT "to start the experiment once the" ON LINE 15
12 PRINT "titrant hits the unknown!" ON LINE 16
13 PRINT "Flip up the "x" switch to stop" ON LINE 20
14 PRINT "the experiment." ON LINE 21
15 WAIT UNTIL INPUT FROM SwW = Pressed
16 CLEAR TEXT SCREEN
17 PRINT "pH" LARGE ON LINE 1
18 PRINT "Temperature" ON LINE 18
19 PRINT "Flip up the "x" switch to stop." ON LINE 21
20 START Timer1 FOR sec
21 PRINT INPUT FROM pH1 LARGE ON LINE 2
22 PRINT INPUT FROM Temp1 ON LINE 19
23 SEND INPUT FROM Timer1 TO COL-A
24 SEND INPUT FROM pH1 TO COL-B
25 SEND INPUT FROM Temp1 TO COL-C
26 IF INPUT FROM SwX = On GOTO 29
27 DELAY 250 millisec
28 GOTO 21
29 CLEAR TEXT SCREEN
30 PRINT "Press any key to return" ON LINE 11
31 PRINT "to the main menu." ON LINE 12
32 STOP
```

FIGURE 40. Program to Obtain pH and Temperature Values for a Timed Titration.

```
1 CLEAR TEXT SCREEN
2 PRINT "This program is used to measure pH and temp" ON LINE 1
3 PRINT "versus volume for an acid/base titration." ON LINE 2
4 PRINT "Enter the total volume of titrant added" ON LINE 4
5 PRINT "when prompted." ON LINE 5
6 PRINT "Make sure you stir the solution well" ON LINE 7
7 PRINT "before you actually enter the value." ON LINE 8
8 PRINT "A pH and Temp will be displayed on the" ON LINE 10
9 PRINT "screen and sent to Cols B and C of the" ON LINE 11
10 PRINT "Spreadsheet. Total volume will be sent" ON LINE 12
11 PRINT "to Col A." ON LINE 13
12 PRINT "Flip up the "x" switch to stop" ON LINE 20
13 PRINT "the experiment." ON LINE 21
14 PAUSE
15 CLEAR TEXT SCREEN
16 PRINT "pH" LARGE ON LINE 1
17 PRINT "Temperature" ON LINE 18
18 PRINT "Flip up the "x" switch to stop." ON LINE 21
19 SEND INPUT FROM Kbd TO COL-A
20 SEND INPUT FROM pH1 TO COL-B
21 SEND INPUT FROM Temp1 TO COL-C
22 PRINT INPUT FROM pH1 LARGE ON LINE 2
23 PRINT INPUT FROM Temp1 ON LINE 19
24 SEND INPUT FROM COL-A TO GraphX
25 SEND INPUT FROM COL-B TO GraphY
26 IF INPUT FROM SwX = On GOTO 28
27 GOTO 19
28 CLEAR TEXT SCREEN
29 PRINT "Press any key to return" ON LINE 11
30 PRINT "to the main menu." ON LINE 12
31 STOP
```

FIGURE 41. Program to Obtain pH and Temperature Values for a Manual Titration.

into prime examples of how the system allows students to participate in the process of science. The quality of data and ease-of-use of the system appear superlative to other efforts at computer-interfaced titrations (38,67,100-102). In addition to acid/base titrations, the MSU lab interface system can be used to perform potentiometric titrations by using the pH input as millivolt input.

Colorimetry

The area of colorimetry provides opportunities for many different experiments using the MSU lab interface. Colorimetry (as applied to visible radiation) and spectrophotometry (as applied over a wider wavelength region) are experimental techniques widely used in analytical chemistry to determine the concentration of substances in solution. In the literature, the terms colorimetry and spectrophotometry are often used interchangeably. These methods are based upon the measurement of the intensity of the color of a solution, or how much light a solution will absorb.

A fundamental law regarding the amount of incoming light absorbed by a sample is known as Beer's Law (103). Beer's Law states that the amount of light absorbed by a sample (A) is proportional to the amount (concentration, C) of absorbing species in the sample, or

$$A = kC.$$

The above formula is the simplest form of Beer's Law. The k is a constant which depends on the path length through the sample and the nature of the absorbing specie. The above equation shows that the measurement of the absorbance A of a sample allows you to find the concentration of the light-absorbing specie.

As we've seen in Chapter 5, a CdS photocell could be used to measure the intensity of light passing through a sample. The photocell measures current in relation to the amount of incident light. For the MSU lab interface, a simple colorimeter was built which contains three (some versions contain only two) light-emitting diodes (LEDs) as the light sources and a CdS photocell as the detector. The red, yellow, and green LEDs provide light at 626, 585, and 569 nm, respectively, over a relatively narrow band. A diagram of the box colorimeter is shown in Figure 42. The colorimeter, however, does not measure the amount of light absorbed by the sample. It measures the amount of light making it *through* the sample. The fraction of light going through the sample is known as the *transmittance*. Beer discovered that the

transmittance of light through a sample decreases exponentially as the concentration
increases linearly. Beer's Law can now be expressed in mathematical terms with
respect to what the colorimeter can actually measure.
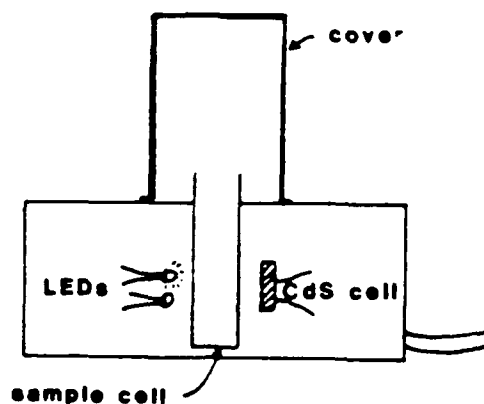
FIGURE 42. Cut-away View of the Colorimeter Box.

Beer's Law is illustrated in Figure 43, where I_o represents the intensity of light
through the sample *without* an absorbing specie and I represents the intensity of light
through the sample *with* an absorbing specie. In terms of what the colorimeter can
measure, these I values are transmission currents.
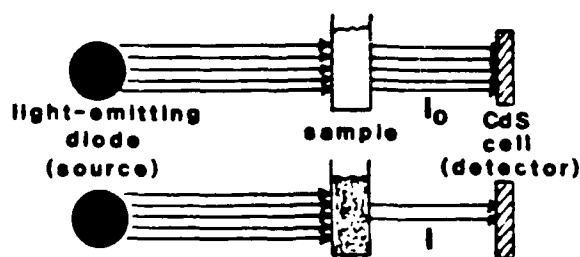
FIGURE 43. Principle Behind Beer's Law.

Based on Figure 43, Beer's Law can be rewritten as follows:

$$A = \log (I_o/I)$$

$$\log(I_o/I) = kC$$

From the form of the previous equation, one can see that a plot of absorbance $(\log(I_o/I))$ versus the concentration of various solutions should yield a straight line with a slope of k if Beer's Law holds. There are a number of limitations to Beer's Law. Beer's Law is *strictly* valid at only low (<0.01 M) concentrations; therefore, *some* deviations can be expected at higher concentrations. For the general chemistry laboratory, Beer's Law is typically applied to solutions of 0.2 M concentration or greater with success. Beer's Law also assumes a monochromatic light source is used. In reality, the LEDs of the colorimeter emit a band or range of wavelengths rather than a single wavelength. If the constant k in the previous equations happens to vary in this band of wavelengths, Beer's Law will fail. In summary, the colorimeter has design limitations which prevent Beer's Law from being followed *exactly*. However, as you will see in the following examples, the colorimeter measurements follow Beer's Law quite well and can be used for quantitative experiments.

Quantitative Light Absorption

One of the most common experiments in general chemistry involving Beer's Law is creating a calibration curve based on the absorbance of known concentrations of solutions, then using that curve to determine the concentration of unknown samples containing the same absorbing specie. The following example, using cobalt(II) chloride, was used as a final project in Chem 125/135 and as part of a laboratory experiment in Chem 136.

A solution of cobalt(II) chloride has a reddish-purple color. Therefore, the green LED is the best choice to use as a light source on the box colorimeter. In this example, I used eight known concentrations of Co^{2+}, ranging from 0 M (distilled water "blank") to 0.300 M. Figure 44 shows a plot of the raw, transmission current data versus the concentration of the sample. The program to obtain the data is relatively straight forward and one that students can certainly be expected to design. The current values on the y-axis range from about 172 microamps down to about 92 microamps.
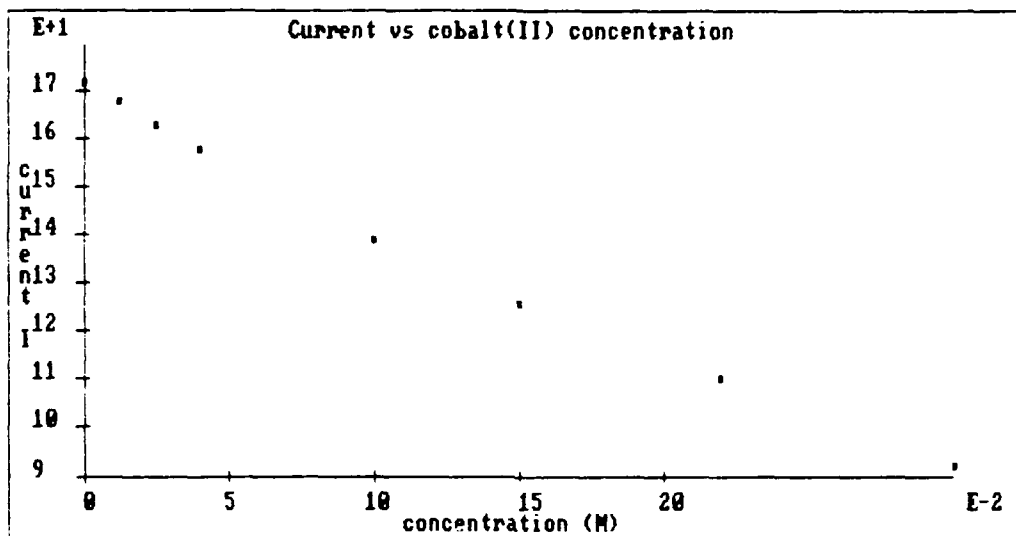
FIGURE 44. Transmission Currents for Co^{2+} Calibration Curve.

As mentioned previously, it is not the transmission currents which are the most meaningful information. On most spectrophotometers, the percent transmittance can be read. From the data in Figure 44, the transmittance could be obtained simply by relating the transmission currents for all the samples to the transmission current for the blank (0 M) sample; that is, the blank is said to have a 100% transmittance.

Performing this operation on the **Spreadsheet** takes about 15 seconds, from typing in the formula to obtaining the results. The transmittance calculation (I/I_o in Column D) and plot (I/I_o versus concentration) are shown in Figure 45. The concentration values in Column A of the **Spreadsheet** were entered via the keyboard as the program was running. The transmission currents (Column B) were automatically obtained and stored by the interface system on the user's command. In analysis Column D, the transmission currents in Column B were converted into transmittance values by dividing all of the currents by the current for the blank (b/171.6).

Column Setup	Acquisition	Analysis	Graph	Stats	Print	File	Quit

Data Acquisition				Data Analysis		EXIT
No.	A: conc.(M)	B: current	C	D: b/171.6	E: log(1/d)	F
1	0.0000	171.6000		1.0000	0.0000	
2	0.0125	167.7000		0.9773	0.0100	
3	0.0250	162.4000		0.9464	0.0239	
4	0.0400	157.4000		0.9172	0.0375	
5	0.1000	138.6000		0.8077	0.0928	
6	0.1500	125.4000		0.7308	0.1362	
7	0.2200	110.3000		0.6428	0.1919	
8	0.3000	91.8000		0.5350	0.2717	

Exit to DOS
1   2   3   4 Grid   5Graph   6D Row   7I Row   8   9 Help 10 Quit
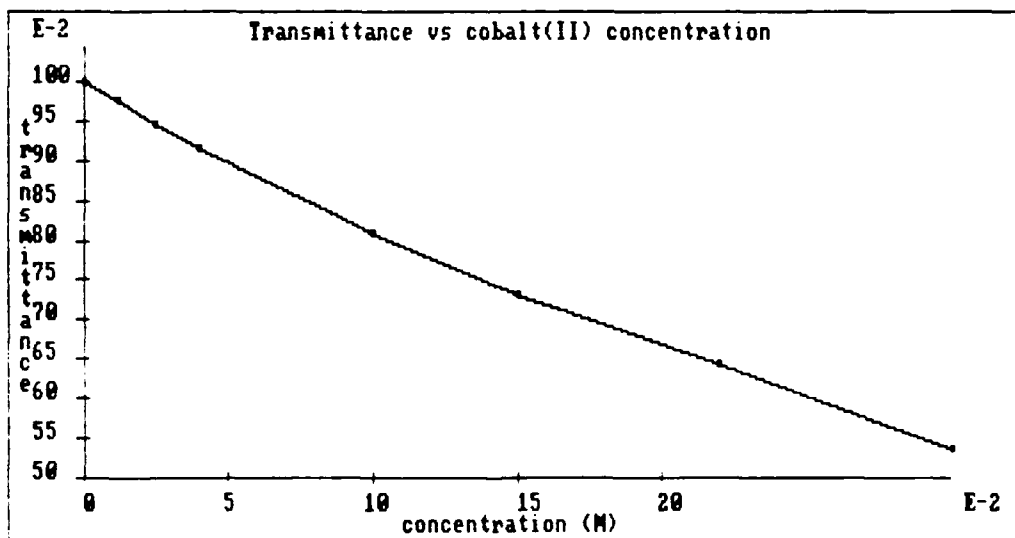
FIGURE 45. Spreadsheet Calculations and Graph for the Transmittance
of the Co^{2+} Samples.

As can be seen in Figure 45, the transmittance versus concentration plot curves downward slightly; it is not linear. The deviation from linearity can be seen much more clearly if a least squares line is drawn through the data points in Figure 45. This is shown in Figure 46. Students can perforr such a linear regression analysis on their data in about 15 seconds. Notice that the equation for the line is given at the top of the graph, as well as a correlation coefficient. A correlation coefficient of +1 or -1 corresponds to a perfect fit of the data to the equation for the line. The linear regression option in plotting is a very powerful tool for "function finding"; that is, performing calculations and functions on the data and determining whether the relationship is becoming more linear. The "y = mx + b" line equation given with the graph in Figure 46 is especially useful when creating calibration graphs and determining experimental values from the slope or intercept of the data.
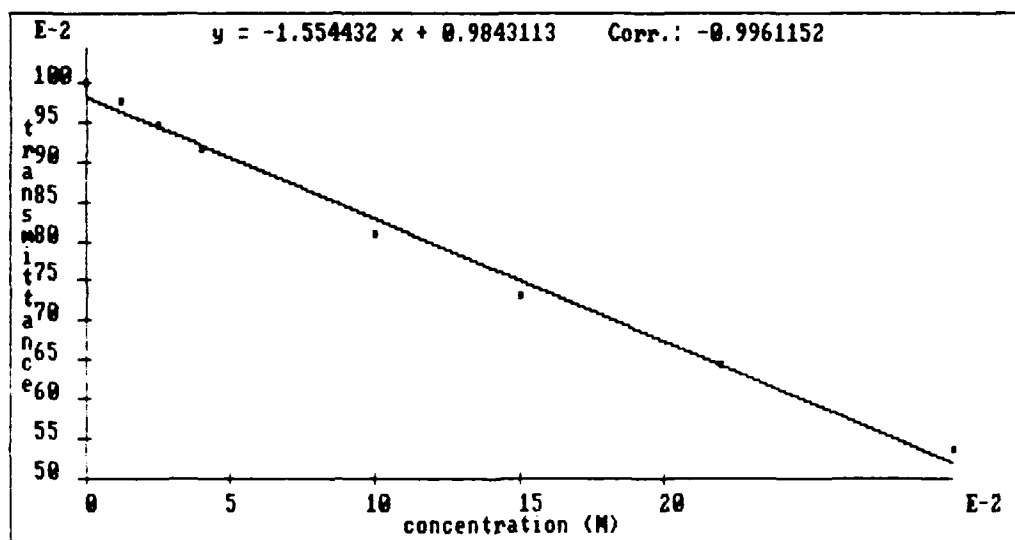
FIGURE 46. Linear Regression Analysis of the Transmittance Data.

Beer's Law states that the concentration of the specie is directly related to the absorbance, *not* the transmittance. From the previous equations, the absorbance is simply equal to $\log(I_o/I)$, which is the same as $\log(1/T)$, where T is the transmittance. The conversion of the transmittance data to absorbance data (see Column E of Figure 45) takes about 15 seconds, and a re-plot takes about another 15 seconds. The absorbance versus Co^{2+} concentrations least squares plot is shown in Figure 47. Note that the correlation coefficient (0.9996104) is considerably closer to a straight line fit than that for the transmittance plot (-0.9961152). Figure 47 can now be used directly to determine the concentrations of unknown samples based on their absorbance. Alternatively, students can use the equation for the line given on their graph to determine the unknown concentrations. This Beer's Law example vividly shows the versatility and speed of the **Spreadsheet** data analysis package. Students can quickly and easily perform mathematical analysis on their experimental data and plot the information. More time is available to study and use the experimental data. The mathematical principles behind Beer's Law come alive on the monitor!
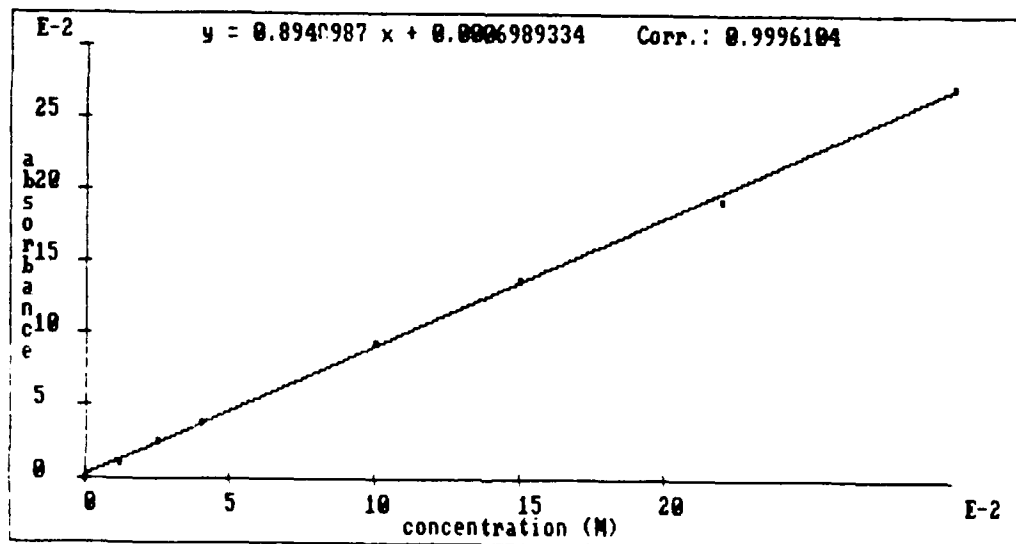
FIGURE 47. Beer's Law: Absorbance Versus Concentration for Co^{2+} Solutions.

The experiment program to obtain the transmission current data is very straightforward and is designed by students as part of the Chem 125/135 final projects. A user-friendly version of the program used to obtain the data in Figure 44 is shown below in Figure 48. The measurement lines are the line that sends the concentration values to the **Spreadsheet** (Line 16) and the lines that read the transmission currents and send the values to the data file (Lines 27 and 30).

```
1 CLEAR TEXT SCREEN
2 PRINT "This experiment allows you to determine the" ON LINE 5
3 PRINT "relationship between the concentration of a sample" ON LINE 6
4 PRINT "and the light transmitted through a sample." ON LINE 7
5 PRINT "Your colorimeter must be properly hooked up to" ON LINE 9
6 PRINT "the lab interface." ON LINE 10
7 PRINT "The blue and yellow plugs go to DAC1 and I1." ON LINE 12
8 PRINT "The other plugs are attached to LED-A," ON LINE 14
9 PRINT "depending on which color LED you wish to" ON LINE 15
10 PRINT "use as your light source." ON LINE 16
11 PAUSE
12 SEND 1700.000000 TO DAC1
13 CLEAR TEXT SCREEN
14 PRINT "Input the molar concentration when prompted." ON LINE 20
15 PRINT "This value will go to Col-A of a data file." ON LINE 21
16 SEND INPUT FROM Kbd TO COL-A
17 CLEAR TEXT SCREEN
18 PRINT "After pressing 'return', a current will appear" ON LINE 4
19 PRINT "which indicates the relative amount of light" ON LINE 5
20 PRINT "passing through the sample." ON LINE 6
21 PRINT "Press the 'F' key to send the value on the screen" ON LINE 10
22 PRINT "to Col-B of a data file." ON LINE 11
23 PAUSE
24 SEND On TO LED-A
25 PRINT "CURRENT" LARGE ON LINE 1
26 PRINT "Press the 'F' key to take data." ON LINE 20
27 PRINT INPUT FROM I1 LARGE ON LINE 2
28 IF INPUT FROM LastKey = 102.000000 GOTO 30
29 GOTO 27
30 SEND INPUT FROM I1 TO COL-B
31 SEND Off TO LED-A
32 CLEAR TEXT SCREEN
33 PRINT "Do you want data on another sample?" ON LINE 1
34 PRINT "Enter '1' for yes, '2' for no." ON LINE 2
35 IF INPUT FROM Kbd = 1.000000 GOTO 13
36 PRINT "Press 'return' to go to the main menu." ON LINE 20
37 STOP
```

FIGURE 48. Experiment Program to Obtain Transmission Current Data.

Kinetics

Unlike a conventional spectrophotometer, the colorimeter attached to the lab interface provides an excellent means of continuously monitoring the progress of certain chemical reactions automatically. The kinetics of reactions which change colors during the progress of the reaction can be studied by monitoring the current on the photocell with respect to time.

One particular reaction ideally suited to such a study is the reaction between molecular bromine (Br_2) and formic acid (HCOOH). This reaction is discussed in some detail in the Chem 136 text by Chang (70), beginning on page 525. For this reason, this reaction was chosen as a project for Chem 136 students. The reaction proceeds as follows:

$$Br_2(aq) + HCOOH(aq) \longrightarrow 2Br^-(aq) + 2H^+(aq) + CO_2(g)$$
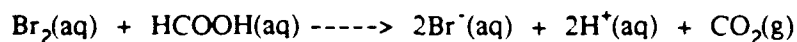
Molecular bromine has a characteristic brown-orange color in aqueous solution, whereas formic acid is colorless. As the above reaction proceeds, the color of the $Br_2(aq)$ fades as it is converted to colorless bromine ion. Therefore, the progress of the reaction can be monitored with a colorimeter using the green LED as the light source. The rate of the reaction can be related to the concentration of the reactants by the following equation:

$$\text{rate} = k[Br_2]^x[HCOOH]^y$$

As seen by the above equation, the rate of the reaction may vary depending upon the concentrations of the reactants. The constant k is a proportionality constant known as the *rate constant*. Since the formic acid is colorless, it is difficult to determine spectrophotometrically how the rate is affected as the concentration of formic acid changes. By using a large excess of formic acid in the reaction, we can

say that the concentration of formic acid does not change; that is, $k[\text{HCOOH}]^y$ is a

constant, which we'll call k'. The rate expression for the reaction now becomes

$$\text{rate} = k'[\text{Br}_2]^x .$$

It is now possible to determine the *pseudo-rate constant* k' and the *order* of the

reaction with respect to Br_2. The *rate* in the above equation can be written in terms of

the depletion of Br_2 as $-d[\text{Br}_2]/dt$. If the reaction is *first order* with respect to Br_2

(i.e. $x = 1$), the above equation becomes

$$-d[\text{Br}_2]/dt = k'[\text{Br}_2] .$$

Upon rearranging terms and integrating with respect to time, this equation becomes

$$\ln([\text{Br}_2]_o/[\text{Br}_2]_t) = k't .$$

By rearranging the terms in this equation, we can obtain an equation in the form of

$y = mx + b$:

$$\ln[\text{Br}_2]_t = (-k')(t) + \ln[\text{Br}_2]_o$$

Therefore, if the reaction is *first order* in Br_2, a plot of $\ln[\text{Br}_2]_t$ versus time will give a

straight line with a slope of $-k'$. Similarly, if the reaction is *second order* in Br_2

(i.e. $x = 2$) the integrated rate expression has the following form:

$$1/[\text{Br}_2]_t = (k')(t) + 1/[\text{Br}_2]_o$$

If the reaction is second order, a plot of $1/[\text{Br}_2]_t$ versus time would yield a straight

line with a slope of k'. Notice from the previous two equations that k' will have

different units depending on whether the reaction is first order (sec^{-1}) or second order

($\text{M}^{-1}\text{-sec}^{-1}$). It is apparent that the MSU lab interface system is an ideal means of not

only obtaining the data, but also for "function fitting" the data to determine the order

of the reaction with respect to bromine.

It is again necessary to use Beer's Law to find the bromine concentrations. The

most useful form of Beer's Law for this experiment is

$$\log(I_o/I) = kC.$$

By measuring a transmission current for a sample without bromine (I_o) and a current

for a solution with a *known* concentration of bromine (I), the value for k can be

calculated. This value is remarkably consistent from colorimeter to colorimeter and has the value of 2.695 M^{-1} for the Br_2(aq) specie using the green LED. With the value of k known, all of the values for bromine concentrations can be determined simply by measuring the transmission currents and I_o.

Figure 49 is a plot of Br_2 concentration versus time for the bromine/formic acid reaction. Three milliliters of ~0.01 M Br_2(aq) were placed into a plastic cuvette, which was subsequently placed in the colorimeter. One drop of 6 M formic acid (which is sufficient excess) was then added to the cuvette and the experiment began. The curve has somewhat of a sawtooth shape due to the fact that the changes in the transmission current quickly become quite small and noisy. The experiment obtained 71 data points in about 75 seconds. It is readily apparent from Figure 49 that the Br_2 concentration decreases rapidly, tailing off towards zero. Carbon dioxide bubbles are also apparent in the cuvette after it is removed from the colorimeter.
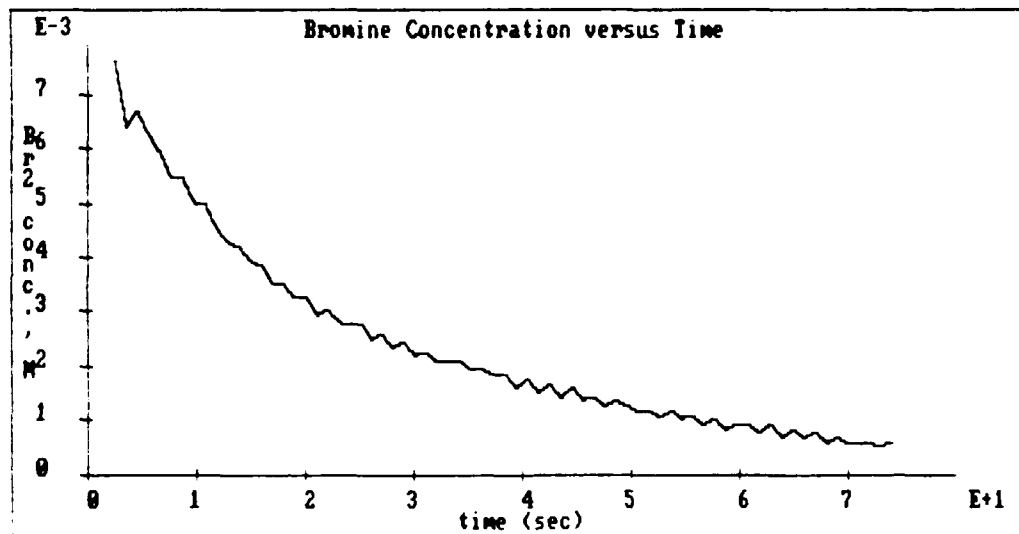
FIGURE 49. Changing Bromine Concentration During the Br_2/HCOOH Reaction.

The **Spreadsheet** provides the ideal tool for quickly determining the order of the reaction. By using the analysis columns to obtain the natural logarithm (ln) of the

bromine concentrations, a plot of ln[Br$_2$] versus time can be rapidly obtained. Figure 50 shows the results of this plot.
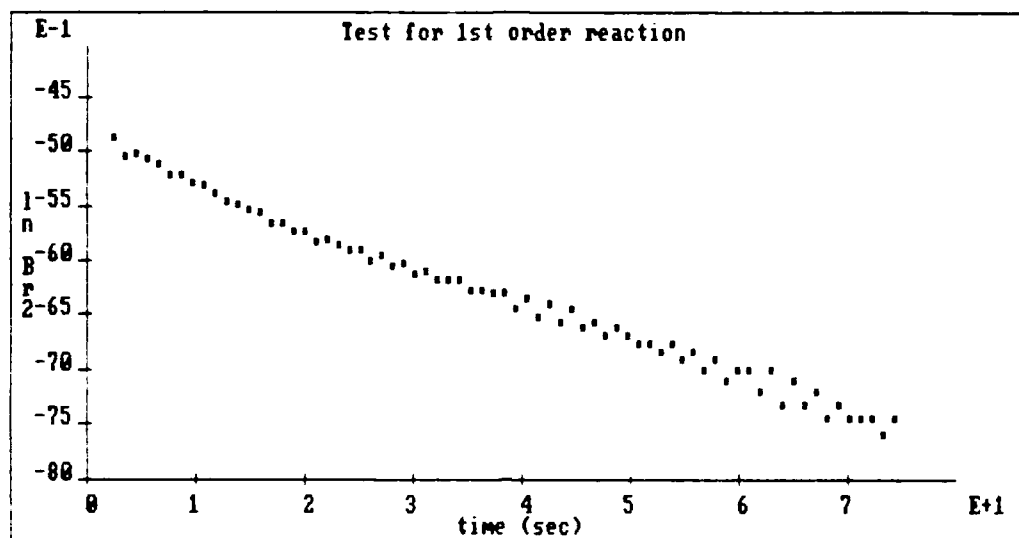
FIGURE 50. Test for a First Order Reaction: ln[Br$_2$] Versus Time.

The results of the linear regression plot of this data is shown in Figure 51. Notice the equation for the line and the correlation coefficient.
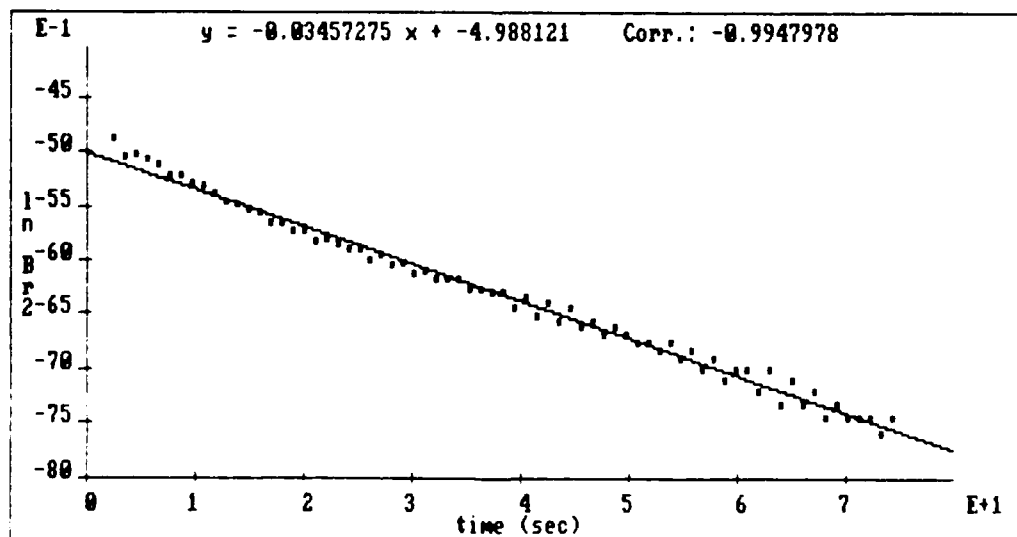
FIGURE 51. Least Squares Plot for First Order Relationship.

From the correlation coefficient, the data appears quite linear. According to

the equation for the line, the reaction has a pseudo-rate constant k' of 0.0346 s^{-1}.

But what if the reaction is second order in bromine? If so, the plot of $1/[Br_2]$ versus

time should give a better straight line than the plot in Figure 51. Figure 52 is a plot of

this function, along with the best-fit least squares line. It is obvious that the data in

Figure 51 is much closer to a linear relationship. Higher order relationships are not

explored in general chemistry; the students would conclude that the bromine/formic

acid reaction is first order with respect to bromine.
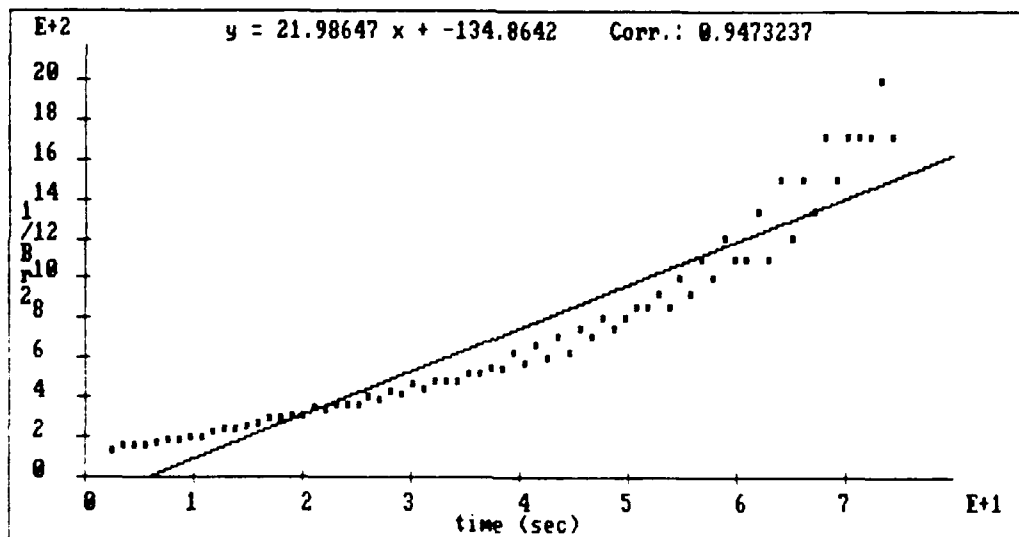
FIGURE 52. Test for Second Order in Br_2 on the Br_2/HCOOH Reaction.

The experiment program designed to obtain this data is closely related to the

thermometric titration program in Figure 26, except current (I1) is being measured

instead of temperature. The program is shown in Figure 53. From a student's

perspective, it is almost identical to the example discussed in Lesson 4 of their

computer manual and detailed in Chapter 5 of this thesis. Lines 26-35 constitute the

measurement loop of the program. Most of the other lines are to make the program

user-friendly.

```
 1 CLEAR TEXT SCREEN
 2 PRINT "This program is used to study the" ON LINE 1
 3 PRINT "kinetics of the bromine/formic acid" ON LINE 2
 4 PRINT "reaction." ON LINE 3
 5 PRINT "The colorimeter should be using the" ON LINE 5
 6 PRINT "GREEN LED." ON LINE 6
 7 PRINT "The program will run for 500 seconds or" ON LINE 8
 8 PRINT "until you flip up the x-switch." ON LINE 9
 9 PRINT "whichever comes first." ON LINE 10
10 PAUSE
11 CLEAR TEXT SCREEN
12 PRINT "Place your cuvette w/3.00 mL of" ON LINE 1
13 PRINT "bromine water into the colorimeter." ON LINE 2
14 PRINT "At the same time you add ONE DROP" ON LINE 4
15 PRINT "of formic acid to the cuvette," ON LINE 5
16 PRINT "press the w-button on the interface box" ON LINE 6
17 PRINT "to start the experiment." ON LINE 7
18 WAIT UNTIL INPUT FROM SwW = Pressed
19 START Timer1 FOR sec
20 CLEAR TEXT SCREEN
21 SEND 1500.000000 TO DAC1
22 PRINT "Replace cuvette with a blank near" ON LINE 18
23 PRINT "the end of the exp. to get a value" ON LINE 19
24 PRINT "for Io (blank current)" ON LINE 20
25 SEND On TO LED-A
26 SEND INPUT FROM Timer1 TO COL-A
27 SEND INPUT FROM I1 TO COL-B
28 PRINT INPUT FROM COL-A LARGE ON LINE 1
29 PRINT INPUT FROM COL-B LARGE ON LINE 2
30 SEND INPUT FROM Timer1 TO GraphX
31 SEND INPUT FROM I1 TO GraphY
32 DELAY 500 millisec
33 IF INPUT FROM Timer1 > 500.000000 GOTO 36
34 IF INPUT FROM SwX = On GOTO 36
35 GOTO 26
36 SEND Off TO LED-A
37 STOP
```

FIGURE 53. Program for the Br_2/HCOOH Kinetics Experiment.

Studying the kinetics of the bromine/formic acid reaction is an excellent example of using the **Spreadsheet** as a "function finder." Complex mathematical relationships can be seen quickly and easily, rather than lost in the tedium of hand-plotting and error-prone calculations. In addition, the experiment for the data acquisition can be designed by the student, making the entire discussion about chemical kinetics in a textbook a real, solvable problem.

Nuclear Chemistry

Nuclear chemistry, or radiochemistry, is also a good subject area for illustrating the capabilities of the MSU lab interface. In nuclear chemistry, one needs accurate, repetitious measurements of isotope activities. In addition, nuclear chemistry is a field in which many experiments are too dangerous for the general chemistry laboratory and are ideally suited for simulation. The MSU lab interface can make isotope activity measurements and simulate them, as well.

Decay as a Random Process

An ordinary Civil Defense Geiger counter displays an analog reading of the radiation rate. However, the MSU lab interface can directly read counts from the Geiger Counter as a measure of activity. The measurement of counts makes the interface an excellent tool for studying the randomness of radioactive decay from a sample where most of the isotopes have very long half-lives.

By far the major radioactive isotope in naturally occurring uranium compounds is U^{238}. U^{238} has a 99.274% abundance and a half-life of 4.51×10^9 years (104). Thus, the radioactivity of a sample of a naturally occurring uranium compound such as uranyl nitrate ($UO_2(NO_3)_2 \cdot 6H_2O$) can be monitored in an experiment with an insignificant loss of material. By observing the radioactivity of a uranyl nitrate sample, we should observe that the nuclear decay process is indeed random.

For this example experiment, a Geiger tube was place 5 cm above a ~1 g sample of uranyl nitrate. The uranyl nitrate was covered with plastic wrap and attached to a 3 X 5 card. The plastic wrap was sufficient to block the alpha radiation. The Geiger tube, then, would measure beta and gamma radiation. To study the randomness of the decay process, the number of counts (nuclei disintegrations) were measured over a 30-second time period. This count was repeated at the 5 cm distance a total of 20 times. Students performed this same experiment as a "canned" experiment in Chem 136. Figure 54 shows the randomly scattered counts data.

E+1   Fluctuations in decay rates

108

106

104

C 102
o
u 100
n
t 98
s

96

94

92

90

0   2   4   6   8   10   12   14   16   18   E+0
                            Trial number
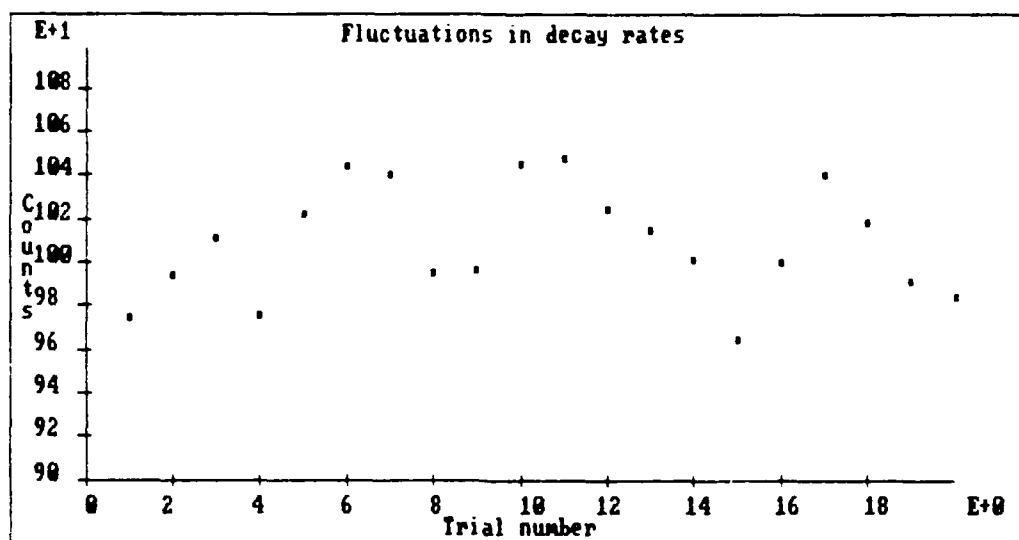
FIGURE 54. Counts from a Uranium Sample Over a 30-Second Time Period for Twenty Experiments.

Although the data in Figure 54 *appears* randomly scattered, students can verify this by using the statistics options in the **Spreadsheet**. The range of a random distribution is such that 66.7% of the values obtained in a random count will be within + or - one standard deviation of the mean. In addition, 95.5% of the values should be within +/- two standard deviations of the mean. The **Spreadsheet**, by using the **Stats** option, will quickly calculate the mean and/or standard deviation of any column of data. For the above data, the number of counts ranged from 964 to 1048. The mean number of counts was 1009, with a standard deviation of 25.8. Figure 55 shows the same data as shown in Figure 54. In Figure 55, the mean value of counts is shown by the thick black line in the middle of the data. The dashed lines bracket the counts within + or - one standard deviation of the mean. The thin black lines bracket the counts within + or - two standard deviations of the mean. If the data is indeed random, students are asked to predict how many of the 20 data points should be *expected* to fall within one standard deviation of the mean. Based on the 66.7% value stated above, one would expect that between 13 and 14 of the count values should be

within one standard deviation of the mean. For this example, there are 12 values within a standard deviation of the mean. Likewise, one should *expect* between 19 and 20 of the count values to be within two standard deviations of the mean. In this example, all twenty values are within two standard deviations.
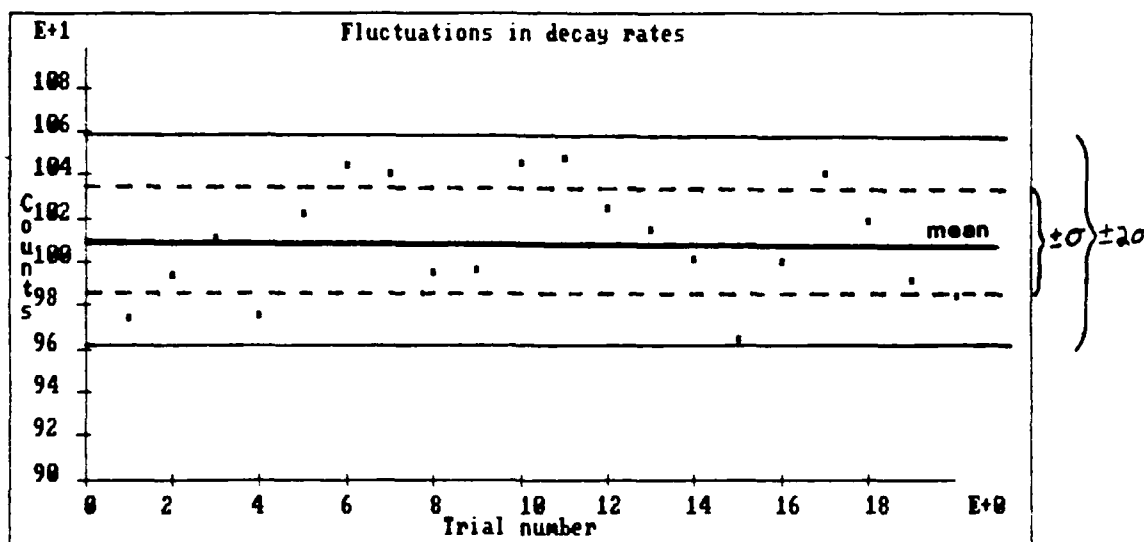
FIGURE 55. Counts Data with Lines Indicating +/- One and Two Standard Deviations of the Mean.

This is an excellent exercise in using statistics to show how radioactive decay is a random process. Statistics can be easily applied to the data students obtained just minutes before using the MSU lab interface system.

Beta Radiation

In a similar experiment, students can study the penetrating ability of beta radiation. This was done as a final project in Chem 125/135 during the Autumn 1989 quarter and as a regularly scheduled experiment in Chem 125 and Chem 136 during the Winter 1990 quarter.

Using the same experimental set-up described previously, students investigated the penetrating power of beta radiation by measuring the absorption of radiation from

the uranium sample by thin index cards and sheets of aluminum foil. Successive numbers of cards were placed between the source and the Geiger tube and the number of counts over a 30-second time period were measured with the lab interface. Because of the 30-second time intervals for data acquisition, the design of the program can be a challenge for students. Figure 56 shows a user-friendly version of this experiment. Lines 18-26 are the true "working lines" of the program.

```
 1 CLEAR TEXT SCREEN
 2 PRINT "In this experiment, you will attempt to" ON LINE 1
 3 PRINT "find out how much material must be used" ON LINE 2
 4 PRINT "to absorb or stop the beta radiation" ON LINE 3
 5 PRINT "from a radioactive sample of uranium." ON LINE 4
 6 PRINT "The program will ask you to input the" ON LINE 11
 7 PRINT "number of cards between the GM tube" ON LINE 12
 8 PRINT "and the source." ON LINE 13
 9 PRINT "Once you've entered that value," ON LINE 15
10 PRINT "the program will count for 30 sec." ON LINE 16
11 PRINT "The number of cards will be entered in" ON LINE 18
12 PRINT "Column A of your Spreadsheet file." ON LINE 19
13 PRINT "The number of counts will be entered in" ON LINE 20
14 PRINT "Column B of your Spreadsheet file." ON LINE 21
15 PAUSE
16 CLEAR TEXT SCREEN
17 PRINT "Enter the total number of cards." ON LINE 20
18 SEND INPUT FROM Kbd TO COL-A
19 CLEAR TEXT SCREEN
20 RESET COUNTER Count1
21 START Timer1 FOR sec
22 PRINT INPUT FROM Count1 LARGE ON LINE 1
23 PRINT INPUT FROM Timer1 LARGE ON LINE 2
24 IF INPUT FROM Timer 1 >= 30.000000 GOTO 26
25 GOTO 22
26 SEND INPUT FROM Count1 TO COL-B
27 SEND INPUT FROM COL-A TO GraphX
28 SEND INPUT FROM COL-B TO GraphY
29 PRINT "Are you finished taking data?" ON LINE 20
30 PRINT "enter a "1" for yes, a "2" for no." ON LINE 21
31 IF INPUT FROM Kbd = 2.000000 GOTO 16
32 CLEAR TEXT SCREEN
33 PRINT "Press any key to return to the main menu." ON LINE 12
34 STOP
```

FIGURE 56. Experiment Program to Determine Beta Radiation Penetration.

In the experiment, the beta radiation will eventually be totally blocked by the index cards, leaving the gamma radiation as the only radiation reaching the detector from the uranium sample. This range of beta penetration through index cards can then be determined by examining the graph of radioactivity versus cards or it can be determined statistically from the counts data. Figure 57 shows a plot obtained using the program in Figure 56. Index cards were placed between the source and the Geiger tube in five card increments. The black arrow shows the point (40 cards) at which the activity has leveled out, indicating only gamma radiation is being observed after that point.
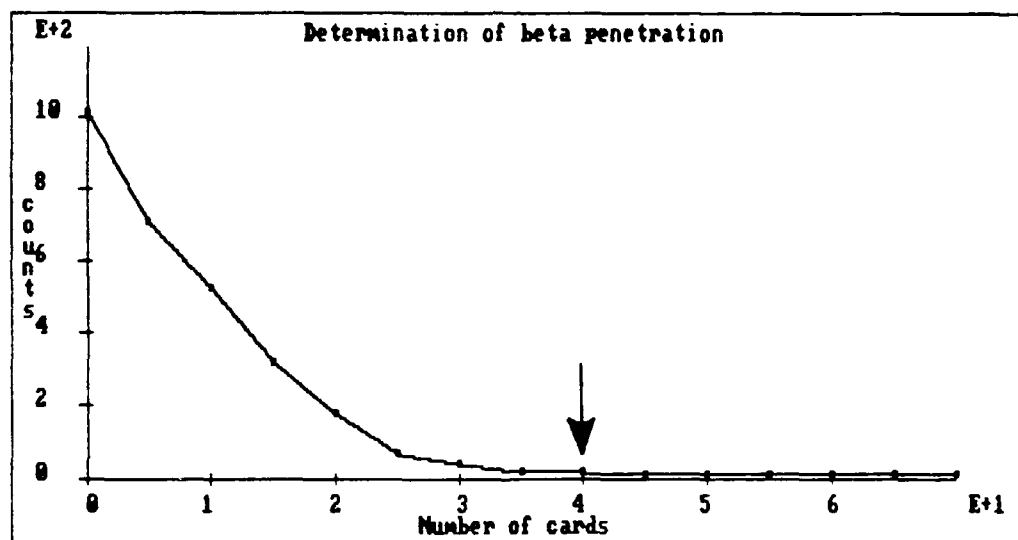
FIGURE 57. Determination of Beta Penetration Limit Through Index Cards. The black arrow indicates the number of cards required for beta blockage.

Since radioactive decay is a random process, the "leveling out" point can also be determined statistically. For the uranium sample, the observation time (30 seconds) is very short compared to the half life. As a result, a single observation of radioactive disintegration rate may be used to estimate the standard deviation (104). The standard deviation may be estimated simply by taking the square root of the observed number of counts for that observation. By taking the square root of all of the counts data for

each placement of index cards, the standard deviation can be calculated for each measurement. For the counts data, when a data point falls within a standard deviation of the point above it, we can consider that the end of the beta particle penetration has been reached. No statistically significant change occurs when additional cards are added beyond this point. Applying this statistical method to the data shown in Figure 57, one also concludes that complete beta blockage occurs at 40 index cards. This method is illustrated in Table 9 below, using the same data as shown in Figure 57.

Table 9. Determination of Beta Radiation Penetration Using the Standard Deviation Method.

Number of Cards	Counts (C) per 30 sec.	Square Root of Counts ($\sqrt{C}$)
0	1013	31.8
5	712	26.7
10	530	23.0
15	318	17.8
20	184	13.6
25	72	8.49
30	41	6.40
35	20	4.47
40	17	4.12
45	12	3.46
50	13	3.60
55	12	3.46
60	9	3.00
65	12	3.46
70	10	3.16

Before we leave the index card experiment, it is important to point out another graphing option in the **Spreadsheet**. Figure 58 is a bar graph representation of the data in Figure 57 and Table 9. With a small number of data points, the bar graph clearly shows the downward-stepping trend of detectable radioactivity as the number of index cards becomes greater. The index cards experiment is another powerful

example of how easily mathematical and statistical concepts can be integrated into a

chemistry laboratory. This is a very challenging and rewarding experiment for

students to design and discover on their own.
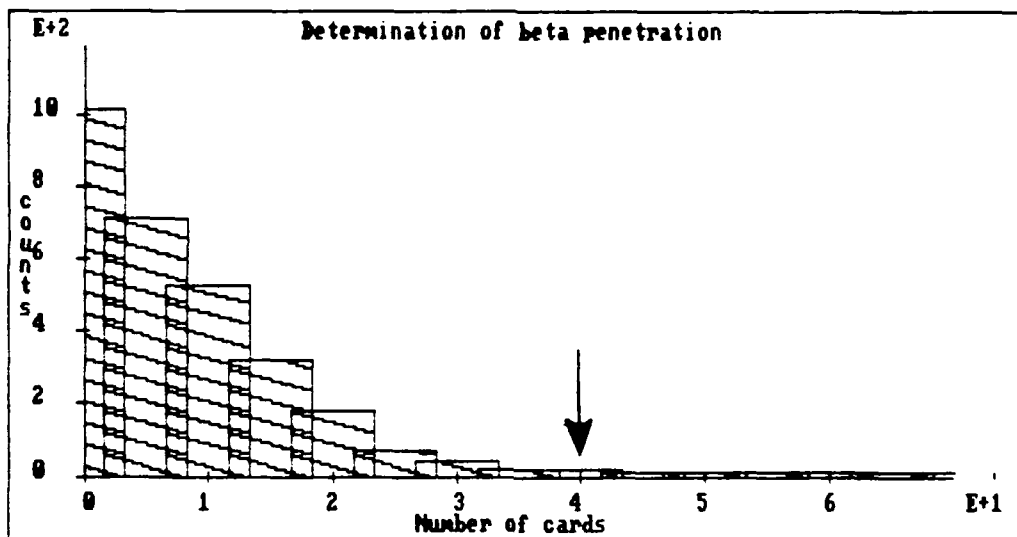
FIGURE 58. Bar Graph of the Beta Penetration Data Using Index Cards.
The arrow indicates the number of cards required for beta blockage.

Determining Half Lives

The rate at which a radioactive element decays depends primarily on two

factors: (1) the stability of the radioactive nuclei, and (2) the number of nuclei

present. These factors make radioactive decay subject to the first order rate law

described with the bromine/formic acid reaction. If N_o represents the decay rate at

time zero and N_t represents the decay rate at some time t, the following expression is

true:

$$\ln(N_o/N_t) = kt$$

From the above equation comes a quantity which is very useful in identifying

isotopes. The *half life* of an isotope is defined as the amount of time required for the

radioactive decay rate of an isotope sample to fall to half. If we let N_t in the above

equation be equal to $1/2 \, N_o$, the equation gives us the following expression for the half life of an isotope:

$$\ln(2) = kt_{1/2} \text{, or}$$

$$t_{1/2} = 0.693/k$$

As can be seen by the above equations, the half life is independent of N_o. Half lives are characteristic of the isotope and can be used to determine the identity of isotopes.

Determining half lives in the general chemistry laboratory can expose students to unnecessary and potentially dangerous levels of radiation. This can be overcome by *simulating* half life experiments. The MSU lab interface can use previously obtained or mathematically generated decay data as a *demonstration file*. The demonstration file is then accessed by an experiment program in real time. The program "thinks" it is obtaining data from the appropriate inputs on the lab interface box, but is really obtaining the data from the demonstration file. No radioactive isotopes are actually used, but the experiment is performed as if "real" data is being obtained. Figure 59 shows the decay curve for Tl-207. The counts data was generated mathematically using the **Spreadsheet** and written into a demonstration file.
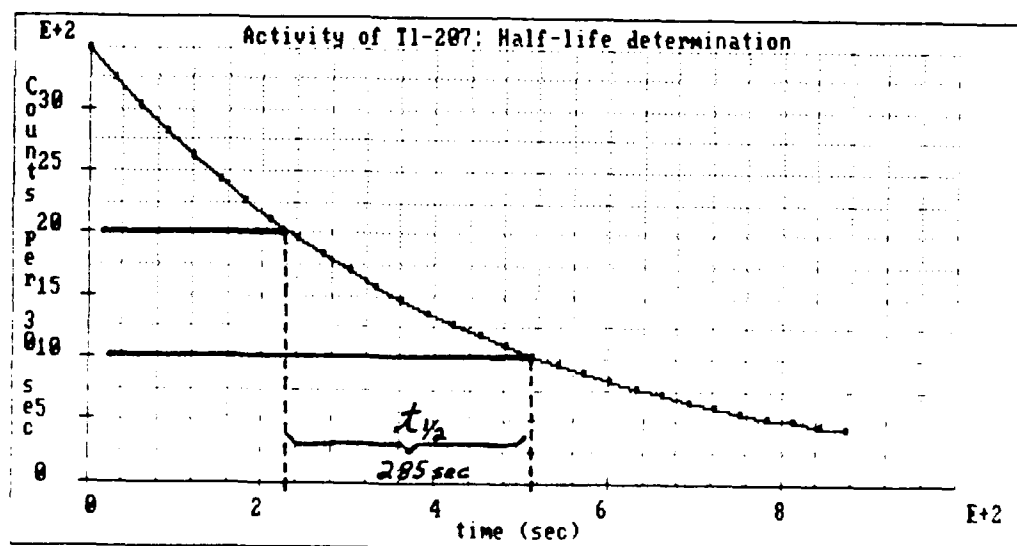
FIGURE 59. Decay Curve for Tl-207.
The actual half life is 288 seconds.

The experiment program in Figure 60 uses real times generated by the lab interface. The half life of the isotope can easily be determined from the graph in Figure 59 by choosing an activity value from the y-axis (2000 counts per 30-sec) and determine the amount of time it takes for the activity to decrease by one-half (1000 counts). Graphically, this half life is 285 seconds; the actual half life is 288 seconds. Students obtain this data for Tl-207 and several other isotopes using demonstration files and the "canned" experiment program in Figure 60.

```
1 CLEAR TEXT SCREEN
2 PRINT "This program measures the decay rate" ON LINE 1
3 PRINT "versus time for an unknown sample." ON LINE 2
4 PRINT "Your goal is to obtain the half-life" ON LINE 4
5 PRINT "of the sample and use the half-life" ON LINE 5
6 PRINT "to identify the sample." ON LINE 6
7 PAUSE
8 CLEAR TEXT SCREEN
9 RESET COUNTER Count1
10 PRINT "Data points will appear" ON LINE 1
11 PRINT "thirty (30) seconds apart." ON LINE 2
12 PRINT "Press the "x" key on the keyboard" ON LINE 8
13 PRINT "to stop taking data." ON LINE 9
14 PRINT "Wait 30 seconds for a response." ON LINE 11
15 START Timer2 FOR sec
16 START Timer1 FOR sec
17 IF INPUT FROM LastKey = 120.000000 GOTO 30
18 PRINT "Counts in last 30 seconds" ON LINE 13
19 SEND INPUT FROM Count1 TO COL-A
20 PRINT INPUT FROM COL-A ON LINE 14
21 PRINT "Time of above count (sec)" ON LINE 16
22 SEND INPUT FROM Timer2 TO COL-B
23 PRINT INPUT FROM COL-B ON LINE 17
24 SEND INPUT FROM COL-B TO GraphX
25 SEND INPUT FROM COL-A TO GraphY
26 PRINT "TOTAL TIME ELAPSED" ON LINE 20
27 PRINT INPUT FROM Timer2 ON LINE 21
28 IF INPUT FROM Timer1 >= 30.000000 GOTO 16
29 GOTO 27
30 PRINT "Press any key to return to" ON LINE 23
31 PRINT "the main menu." ON LINE 24
32 STOP
```

FIGURE 60. Program to Determine Half Lives Using a Demonstration for Counter Input Data.

As seen previously, the integrated first order rate expression also has the form of the following equation:

$$\ln(N_t) = kt + \ln(N_o)$$

Therefore a plot of $\ln(N_t)$ versus time should yield a straight line with a slope of k.

The **Spreadsheet** can be used to quickly obtain the $\ln(N_t)$ values from the counts data and rapidly plot these values versus time. The result of this plot for the Tl-207 data is shown in Figure 61.

Plot with label E-1, equation $y = -0.382396242\ x + 8.161439$, Corr.: -0.9999998; y-axis $\ln(a)$ values 60, 62, 64, 66, 68, 70, 72, 74, 76, 78, 80; x-axis time (sec) values 0, 2, 4, 6, 8, E+2.
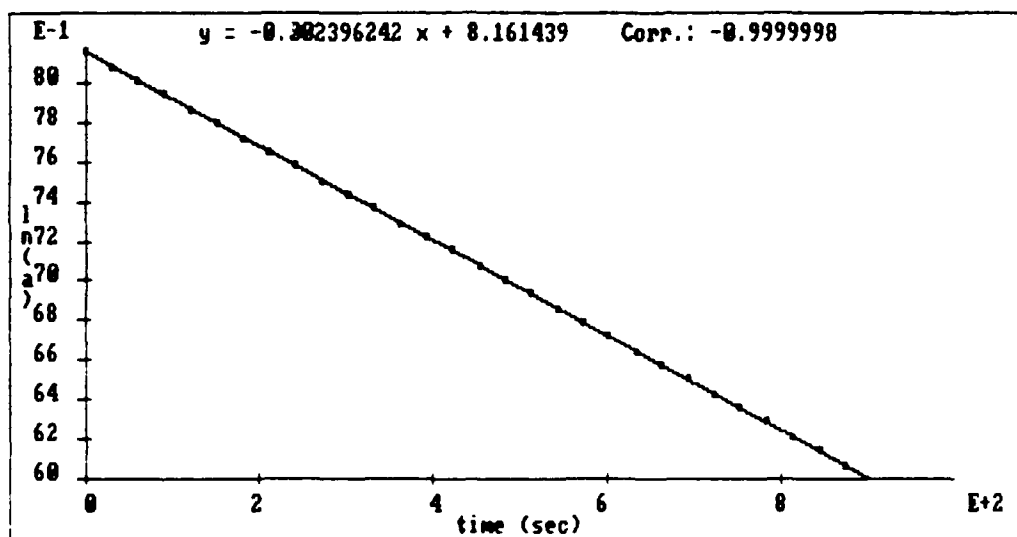
FIGURE 61. $\ln(N_t)$ Versus Time for Tl-207 to Determine Half Life.

The slope of the line is equal to the rate constant k in the equation

$$t_{1/2} = 0.693/k \ .$$

Using the slope from the equation for the least squares line in Figure 61, the half life of Tl-207 is calculated to be 289 seconds.

It is also possible to obtain real half life data by using a long-lived isotope from which short-lived daughter isotopes may be separated. This method is described

by Amend (105) and by Broadbank, Harding, and Patel (106,107). The naturally occurring isotope used is thorium-232 (Th^{232}). Th^{232} has many radioactive daughter products, including Tl^{208}, which is a beta emitter with a half life of 3.1 minutes. The Tl^{208} can be separated from the thorium and the other decay products by dissolving about 5 g of thorium nitrate in 10 mL of 2M HNO_3 and vacuum filtering the solution through a thin bed of ammonium phosphomolybdate (($NH_4)_3PMo_{12}O_{40}$) precipitated on a piece of filter paper (the experimental details are given in Reference 105). The Tl^{208} is adsorbed onto the thin layer of ammonium phosphomolybdate. The half life of the Tl^{208} isotope can then be easily determined by measuring the radioactivity of the sample over 30-second or one-minute time periods for several minutes. The live data from this experiment would appear similar to that in Figure 59.

The nuclear chemistry experiments described in this section underscore the versatility of the MSU lab interface for experiment design, data acquisition, and analysis. The interface also offers the ability to simulate experiments in much the same manner as the videodisc methods of Jones et.al. (25), but without colorful video images.

Many other experiments have been developed and tested for the MSU lab interface. Reference 87 provides perhaps the best source of experiments for a wide variety of subject areas, including earth science, biology, physics, and chemistry. Table 10 is a list of the titles of the experiments in Reference 87. Appendix C is a compilation of user-friendly MSU lab interface experiment programs which were designed to successfully complete these experiments. The programs in Appendix C are given to illustrate the wide variety of experi ts which can be designed with the MSU interface system.

Table 10. Examples of Experiments Designed for the MSU Lab Interface (87).

Earth Science
　　　　Heat Capacity of Materials
　　　　Comparison of Insulation Materials
　　　　Sediment Concentration in Water

Biology
　　　　Hamster Activity
　　　　Human Reaction Rates for Unanticipated and
　　　　　　　Anticipated Events
　　　　Growth Curve of a Yeast Colony

Physics
　　　　Ohm's Law
　　　　Determination of Planck's Constant
　　　　Falling Body Experiment

Chemistry
　　　　Electrochemistry
　　　　Potentiometric and Thermometric Titration of Toilet
　　　　　　　Bowl Cleaner
　　　　Determination of Beta Radiation Penetration

As seen by all the experiment examples, the MSU interface system allows general chemistry students the opportunity to explore many new areas of chemistry previously unavailable to them. Students become involved with the process of science!

CHAPTER 7

RESULTS AND SUMMARY

We have successfully integrated the MSU laboratory interface system into our general chemistry laboratories. Due to the unique features of the hardware and software, our students are able to participate in the process of science by *designing experiments, acquiring data*, and *analyzing the data to draw conclusions*. In a short period of time, our students are able to acquire the conceptual knowledge, lab skills, and computer skills necessary to complete a design project using the MSU lab interface system.

It is important to judge our success not only upon the development our program, but upon the perceptions of our students as well. Although I firmly believe that students should *not* dictate the contents of an introductory course, I also believe that students perceptions are important in determining whether or not a course is meeting its objectives. In general chemistry laboratory courses, "correct" quantitative experimental results are *not* necessarily a measure of student success. Nor is a student's success or failure in a corresponding lecture course indicative of what the student is or is not learning in the laboratory. Therefore, it is difficult if not impossible to quantitatively "measure" whether or not students are doing better in chemistry and learning more chemistry as a result of our approach in the laboratory. Rather, we have concentrated our evaluation efforts on students' perceptions of our approach, *methods*, and *materials*.

Preliminary Evaluations: The First Quarter

Our primary evaluation concerns during the first quarter (Autumn 1989) of full scale implementation of our program were logistical and administrative in nature. The majority of our effort focused on receiving feedback about individual experiments from the lab instructors and the students. For each lab, we solicited written feedback from at least 50 students in both Chem 125 and Chem 135. The laboratory evaluation form included questions about the difficulty of the lab, whether or not the students had enough time to complete the lab, sections of the write-up which were unclear, and the use of the lab in understanding chemical concepts. This feedback was used to modify the labs for the Winter quarter. Each week after a lab was completed, we had a meeting of all laboratory instructors to obtain their verbal feedback about the lab and give them an overview of what was coming the next week.

Since the final project concept was the biggest change from previous years' laboratory courses, we focused the final student critique for the first quarter on the final projects. The following discussion summarizes the results from this voluntary survey in which approximately 800 of the 1200 students enrolled in the general chemistry laboratories responded. The results are summarized below in "binary" form. The survey was not computerized; subsequently, some responses were wishy-washy or unclear. If a response was unclear, it was not included in the tabulation, which is why the total number of responses for each question is not the same.

1. *Did you enjoy doing the final project?* 682 students answered "yes", 104 answered "no".

<div align="center">

87% YES   13% NO

</div>

2. *Do you feel you learned more about experimenting; that is, the "discovery" part of science, as a result of the final project?* 664 answered "yes", 134 answered "no".

<div align="center">

83% YES   17% NO

</div>

3. *The final project was designed to be an independent, "think-on-your-own" research project. Keeping this in mind, do you feel the handout and your instructor gave you enough information to successfully complete the project?* 699 answered "yes", 85 answered "no".

<div align="center">

89% YES   11% NO

</div>

4. *Are you more comfortable with computers as a result of this course?* 619 answered "yes", 142 answered "no". There were many students who answered "no" to this question who also stated that they were already comfortable with computers before having this class. These responses were still considered to be "no" answers; I did not attempt to "interpret" their response.

<div align="center">

81% YES   19% NO

</div>

Binary answers do not tell the whole story. Students were asked to comment on their answers to the previous questions, as well as offer comments and suggestions for improving the final projects. Although the responses were certainly not all favorable, the majority were favorable. Table 11 on the following page contains quotes from some of these comments. It is apparent from these comments and many others like them that students are indeed participating in the process of science *and realizing it! Our approach in the laboratory is allowing students to be participants in the process of science, not spectators.*

Table 11. Comments from the Chem 125/135 Final Project Critique:
Autumn 1989

- "I think the final project is a good idea. It gives you a chance to apply what we've learned about experimenting and chemistry."

- "The project made you think for yourself and use your own experimental design."

- "[It was] interesting to make up our own program and perform [the] experiment on our own."

- "The computer helped a lot."

- "I think the computers are fun and useful to work with."

- "I enjoyed the computers and feel more confident using them now."

- "It [the project] gave me a chance to think out the problems and do the lab the way I thought it should be done."

- "I thought it was interesting to be able to kind of experiment by yourself without the help of our classmates and made us more aware of the experiment and process."

- "I do feel that I have learned more about experimenting as a result of the final project."

- "Through performing this [project] I feel that I learned more about what kind of preparation it could possibly take to perform an experiment for the first time."

- "A lot of work just for one credit!"

- "It [the project] was interesting and very challenging."

- "It was a creative experience."

- "Keep the final projects. They are a good thing."

The Winter quarter of 1990 gave us the opportunity to evaluate our microcomputer-based laboratory concept in all three introductory courses which use the computers: Chem 125, Chem 135, and Chem 136. Having worked out many of the administrative and logistical problems identified in the first quarter, we were ready to obtain a fair evaluation of our approach, methods and materials.

Evaluation of Our Approach, Methods, and Materials

At the end of the Winter 1990 quarter, 190 Chem 125 students, 122 Chem 135 students, and 303 Chem 136 students responded to surveys regarding our approach to using computers in the laboratory, the final projects, and the written materials supporting the laboratory. The Chem 125 and Chem 135 surveys were almost identical, containing eight multiple choice questions plus room for comments. The Chem 136 survey was only slightly different and contained seven multiple choice questions plus room for comments. The results were machine tabulated. In the series of figures that follow, the course number, question number and question as it appeared on the original survey will be used as the figure caption.

As seen in Figure 62, over 75% of the Chem 125 students felt that doing projects in Chem 125 is a good or excellent idea. In further support of the final project concept, Figure 63 shows that 67% agreed that the projects were challenging and 9% strongly agreed that they were challenging.

FIGURE 62. Chem 125, #1: Do you feel that doing projects in Chem 125 is a good idea?
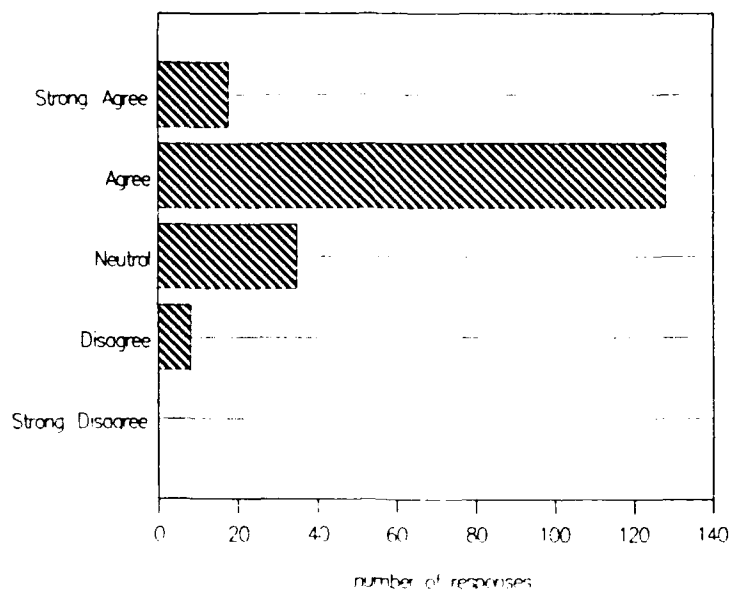
FIGURE 63. Chem 125, #2: The Chem 125 projects were challenging.

Additionally, two-thirds of the students felt that the laboratories leading up to the final projects were also challenging. The results from this question are shown in Figure 64.
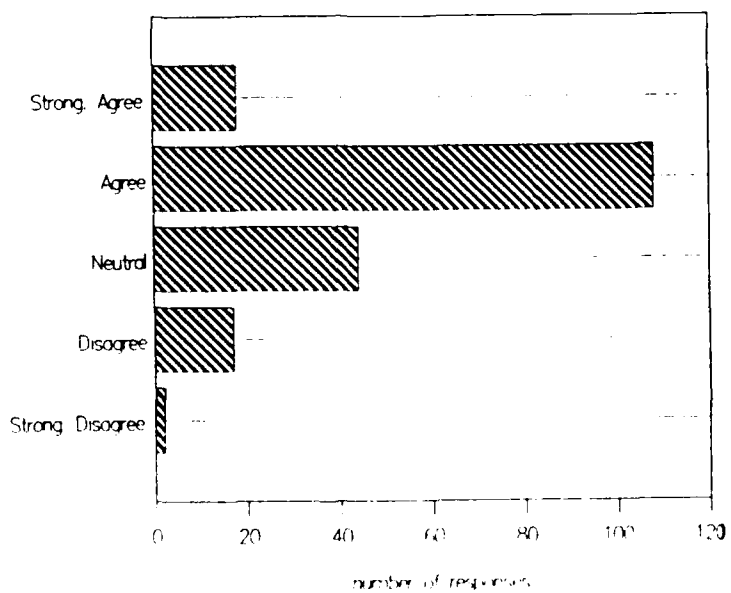
FIGURE 64. Chem 125, #3: The Chem 125 weekly laboratories (not the projects) were challenging.

When asked if the computers were useful for learning chemistry in the laboratory, nearly half (49%) believed the computers were *very* useful and an additional 30% believed the computers were useful. Only a small number of students (7, or 4%) believed that the computers were a hinderance to learning chemistry.

FIGURE 65. Chem 125, #4: In the role of designing experiments, acquiring data, and analyzing data, do you believe that computers are useful for learning chemistry in the laboratory?

Our course also made the introductory Chem 125 students more comfortable with computers. As shown in Figure 66, 57% of the students were more or much more comfortable with computers as a result of the course. Only 3% became less comfortable with computers, which tells us that the software is meeting the very tough standard of being friendly and usable to almost every computer neophyte. This conclusion is also supported by Figure 67 which shows that nearly 90% of the students did not have any difficulty in using the computers in the laboratory. A good deal of the credit for the computers' usability in the laboratory must go to the lab instructors, who were able to answer questions and trouble shoot problems on the spot.

FIGURE 66. Chem 125, #5: Are you more comfortable with computers as a result of taking Chem 125?
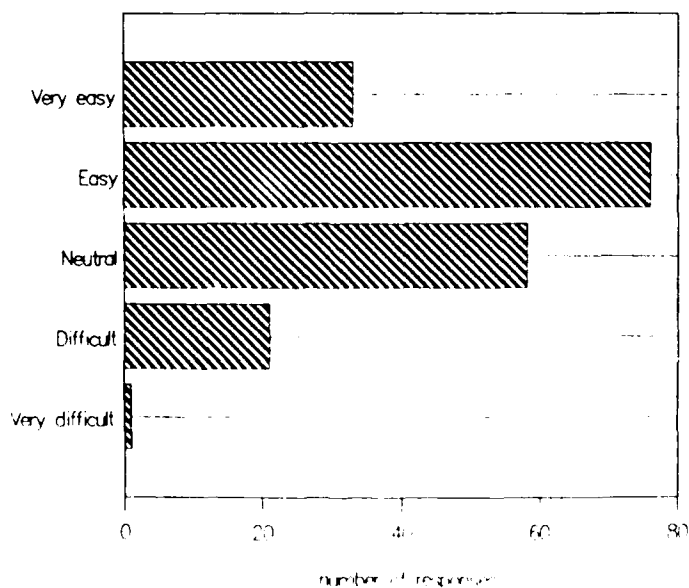
FIGURE 67. Chem 125, #6: Based on your experience in Chem 125, were the laboratory computers easy to use?

The written materials supporting the course were also well received by the students. Before we gave the survey, we decided that our desired responses to the questions presented in Figures 68 and 69 were the *neutral* and *easy* choices. A response of *very easy* may imply that we were writing the manuals at too low of a level to challenge their understanding of the material. Conversely, responses of *difficult* and *very difficult* infer that the material is either poorly written or written at too high of a level for their comprehension. As seen in the following two figures, the responses to the questions regarding the lab book and computer book met with nearly identical results, with nearly 80% of the students rating the books neutral or easy in terms of readability and understandability. Again, students and lab instructors provided many helpful comments the first quarter which allowed us to improve our presentation of the material.
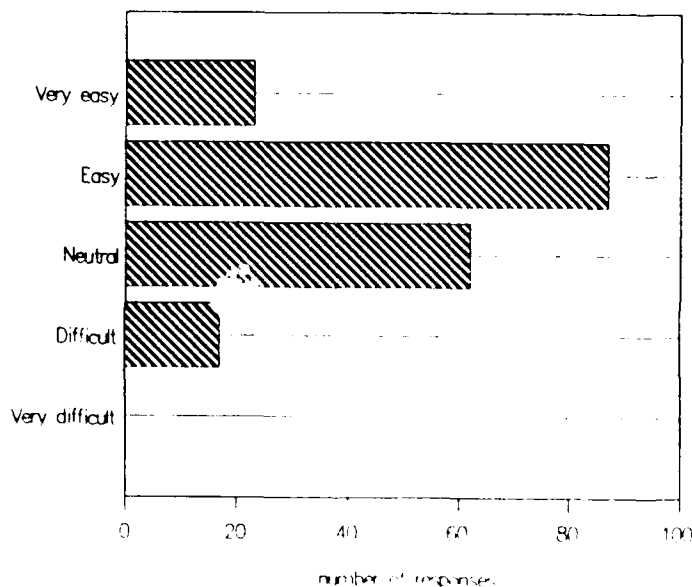
FIGURE 68. Chem 125, #7: Was the Chem 125 lab book easy to read and understand?
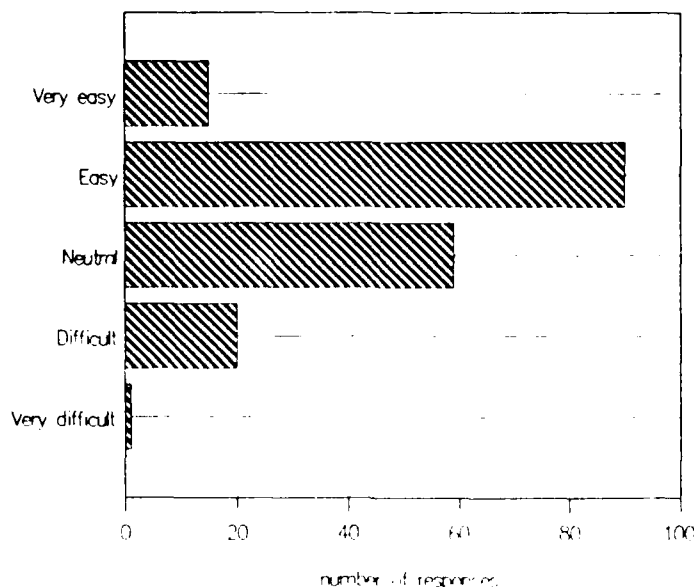
FIGURE 69. Chem 125, #8: Was the book "Using Computers in the General Chemistry Laboratory" easy to read and understand?

The results of the Chem 135 (general chemistry for science and engineering majors) survey were very similar to the Chem 125 survey. From an overall percentage standpoint, the results can be viewed as being slightly more favorable. The similarity of the responses is not surprising. The course was likely their first college chemistry lab, as was the case with Chem 125 students. Even though their lab book was the same as the Chem 125 lab book, the various *levels* available during the experiments provided enough of a challenge to compensate for the Chem 135 students' extra ability. The computer book and computer lessons in the lab were identical to those in Chem 125. Because of the similarity to the Chem 125 results, I will only present the bar graphs as consecutive figures (Figures 70-77) and not give percentage breakdowns of the responses. Of particular note is the Chem 135 response to question #1, regarding their opinion on the final project concept. *Not a single Chem 135 student* felt that doing the final projects was a bad idea; they were *very* supportive of the idea.
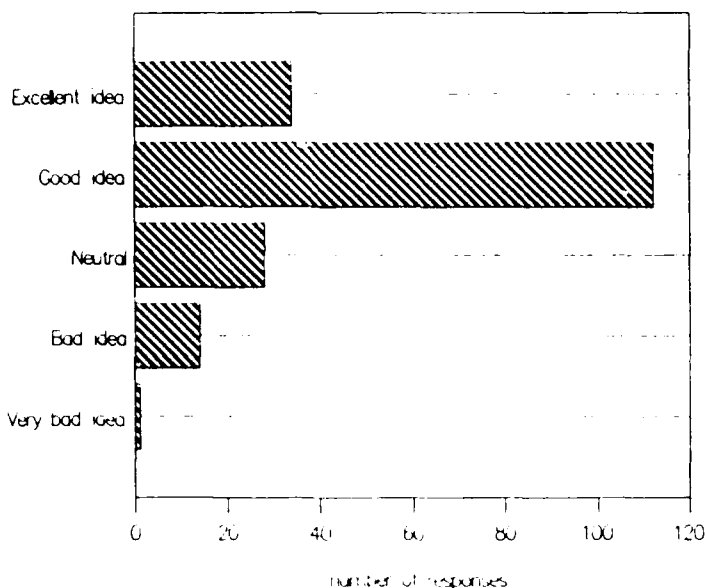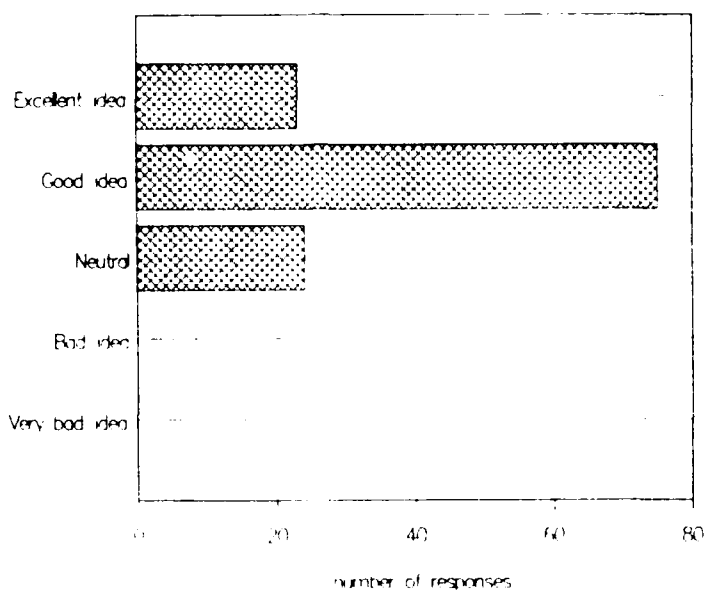
FIGURE 70. Chem 135, #1: Do you feel that doing projects in Chem 135 is a good idea?
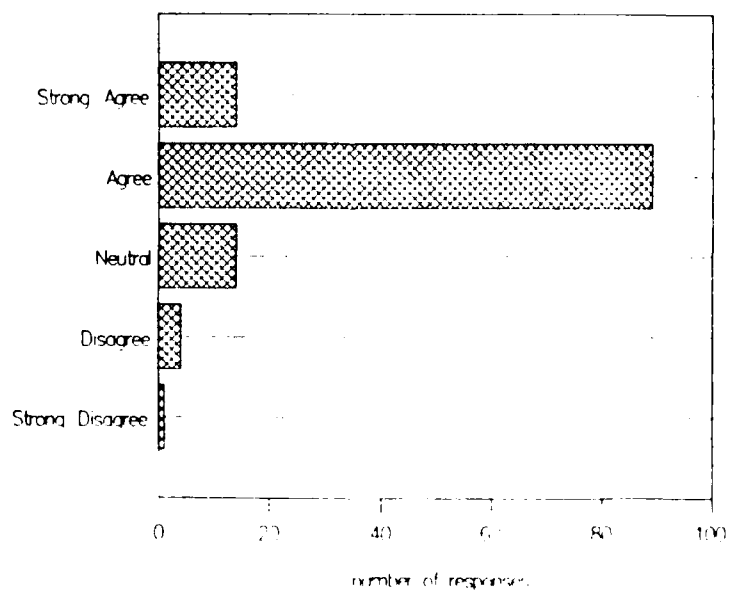
FIGURE 71. Chem 135, #2: The Chem 135 projects were challenging.
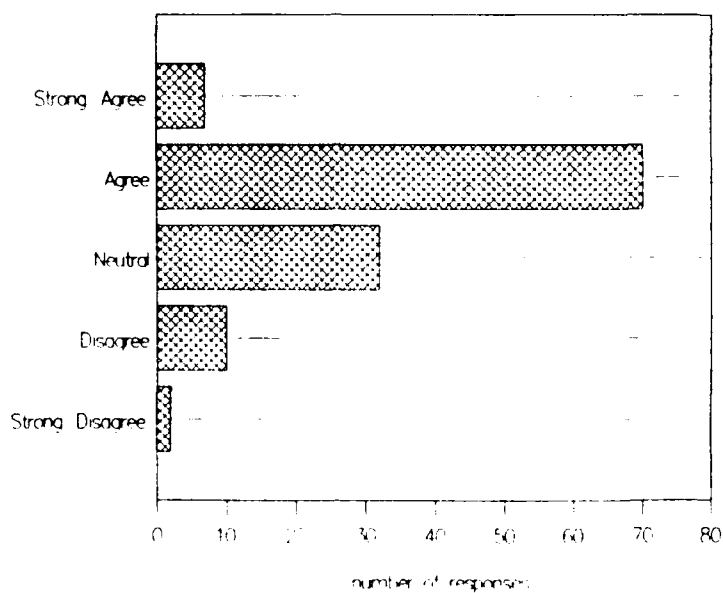
FIGURE 72. Chem 135, #3: The Chem 135 weekly laboratories (not the projects) were challenging.
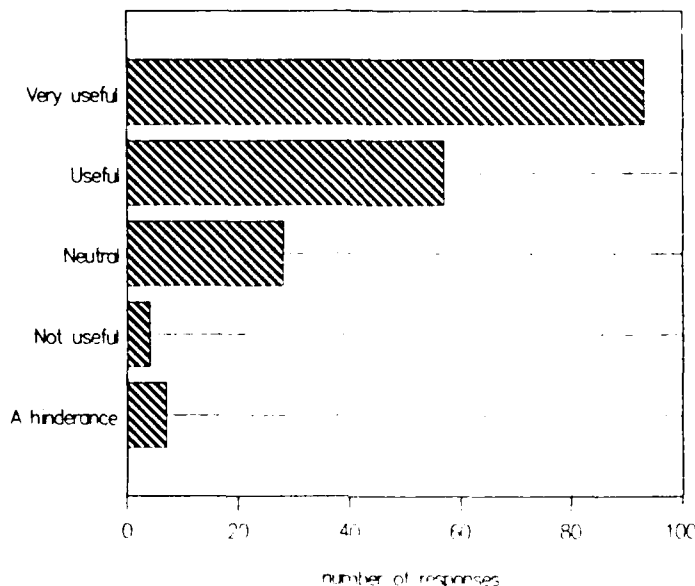

FIGURE 73. Chem 135, #4: In the role of designing experiments, acquiring data, and analyzing data, do you believe that computers are useful for learning chemistry in the laboratory?
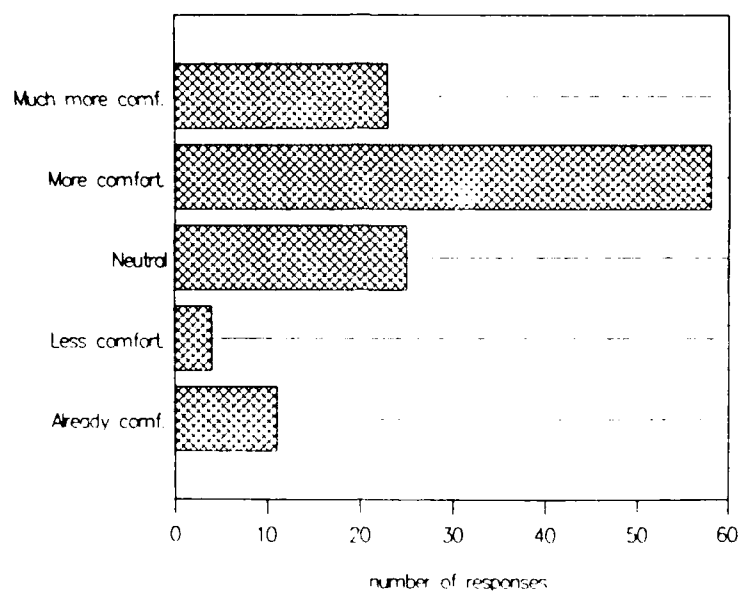
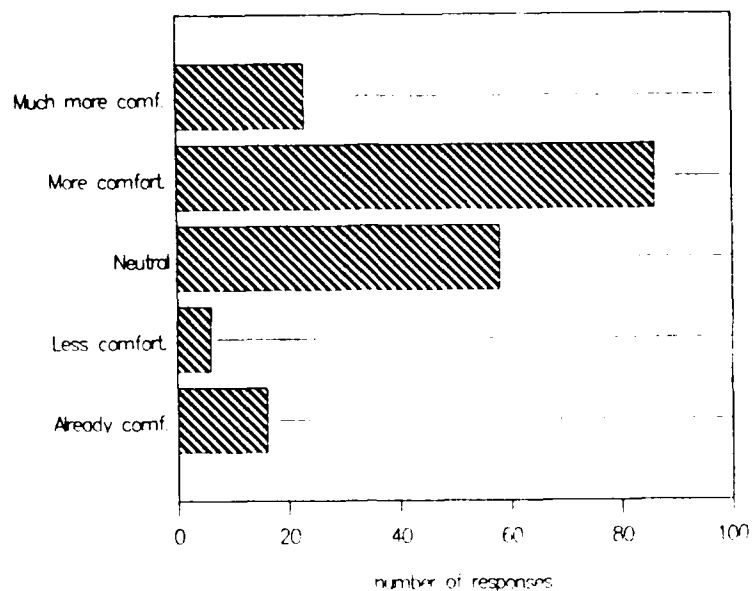**FIGURE 74.** Chem 135, #5: Are you more comfortable with computers as a result of taking Chem 135?
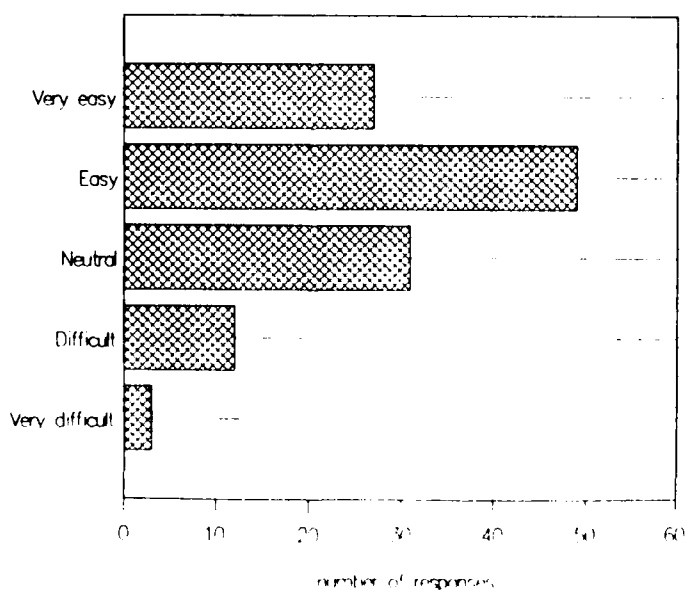
FIGURE 75. Chem 135, #6: Based on your experience in Chem 135, were the laboratory computers easy to use?
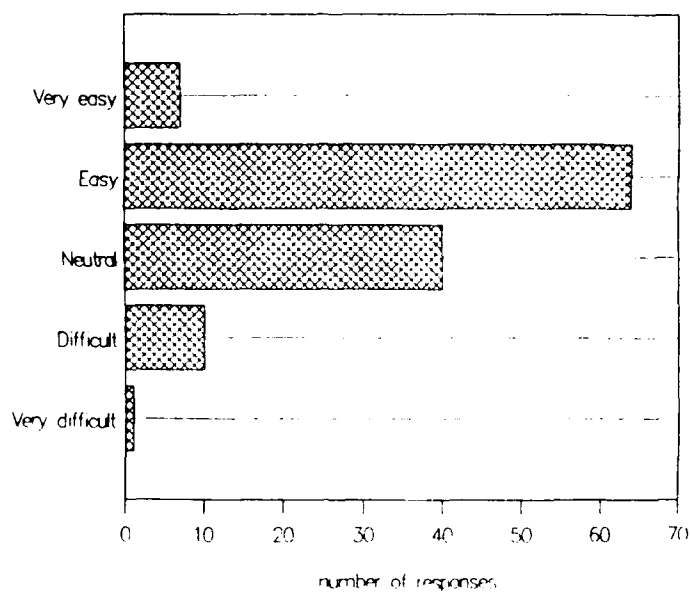
FIGURE 76. Chem 135, #7: Was the Chem 135 lab book easy to read and understand?
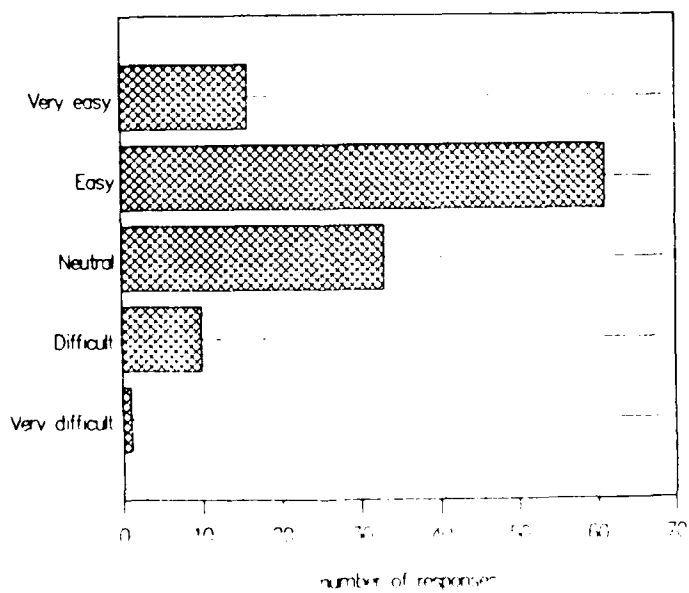
FIGURE 77. Chem 135, #8: Was the book "Using Computers in the General Chemistry Laboratory" easy to read and understand?

The Chem 136 (the follow-on course to Chem 135 for science and engineering majors) survey followed the same format as the Chem 125/135 surveys but did not include a question about the computer book, since they did not use one in this course. The questions pertaining to whether or not the projects and laboratories were challenging are asked relative to what they experienced in Chem 135. As seen in Figure 78, most of the students (61%) felt that the projects were a good or excellent idea in Chem 136. Perhaps there is a greater percentage of *neutral* responses to this question from the Chem 136 students than from the Chem 125/135 students because they have had *two* chances to realize how much work is involved in the projects on *their* part!
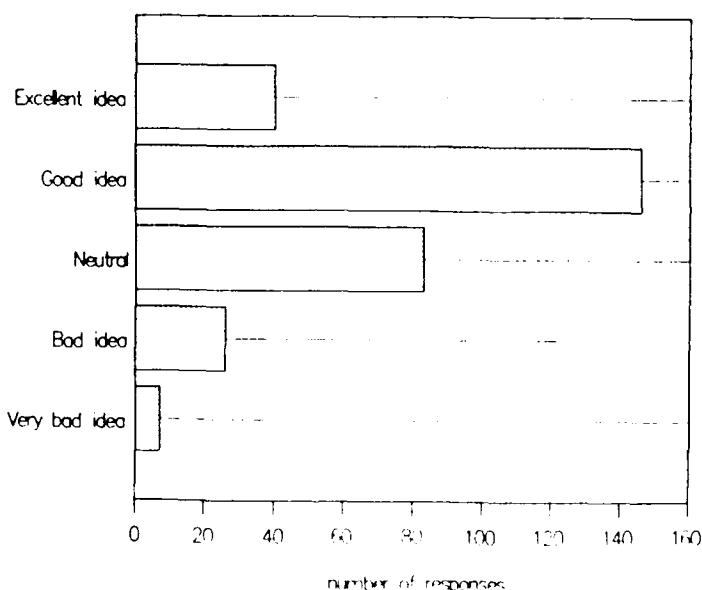
FIGURE 78. Chem 136, #1: Do you feel that doing projects in Chem 136 is a good idea?

The Chem 136 projects were designed to challenge the students' analytical abilities and to emphasize the importance of accurate, quantitative results in an experiment. As Figure 79 shows, 61% of the students perceived that the Chem 136 projects were more challenging that the Chem 135 projects. In addition, 65% of the students believed that the weekly laboratories were more challenging (Figure 80).
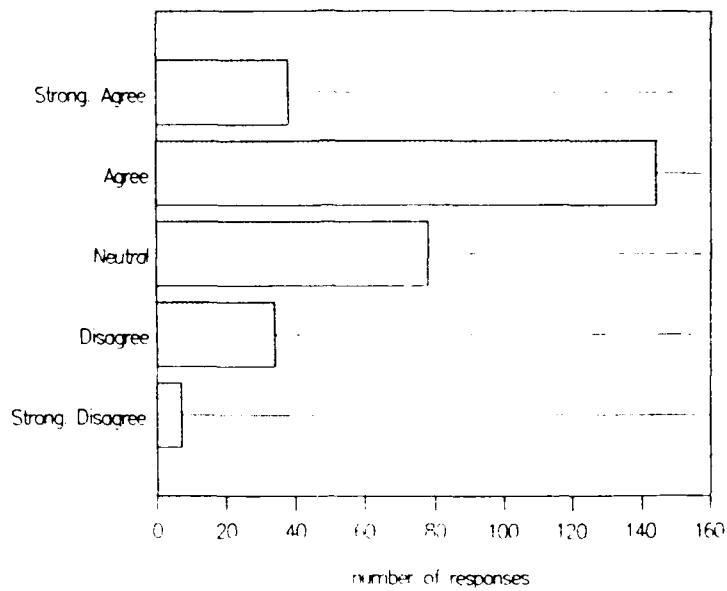
FIGURE 79. Chem 136, #2: The Chem 136 projects were more challenging than the Chem 135 projects.
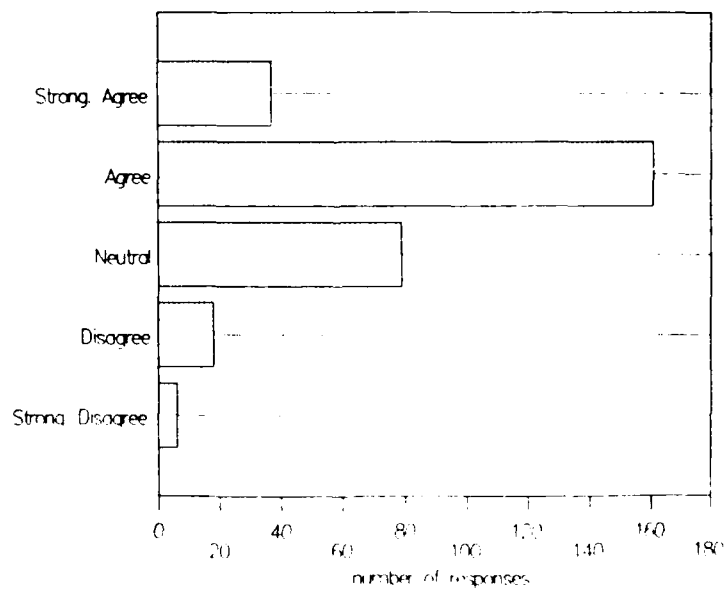
FIGURE 80. Chem 136, #3: The Chem 136 weekly laboratories (not the projects) were more challenging than the Chem 135 laboratories.

Chem 136 students were still very supportive of the usefulness of using computers in the lab. As in Chem 125/135, over 80% believed that the computers were useful or very useful for learning chemistry in the lab (Figure 81). Only a small percentage felt that computers were a hinderance in the lab (5%) or not useful (4%). These percentages are comparable to the Chem 125/135 responses to the same question. A high percentage (47%) felt more or much more comfortable with computers as a result of the Chem 135/136 sequence. It should be pointed out that these students are more likely to have previous computer experience than their Chem 125/135 counterparts simply because of their choice of academic discipline and probable math/science background. This is evident in the greater percentage of *..!ready comfortable* and *neutral* responses than found in Chem 125/135, as seen in Figure 82. Following the trend in Chem 125/135 students, the majority (61%) of Chem 136 students found the laboratory computers easy or very easy to use. The Chem 136 students were basically on their own with the lab computers, having received no additional formal instruction beyond that given in Chem 135 the previous quarter.
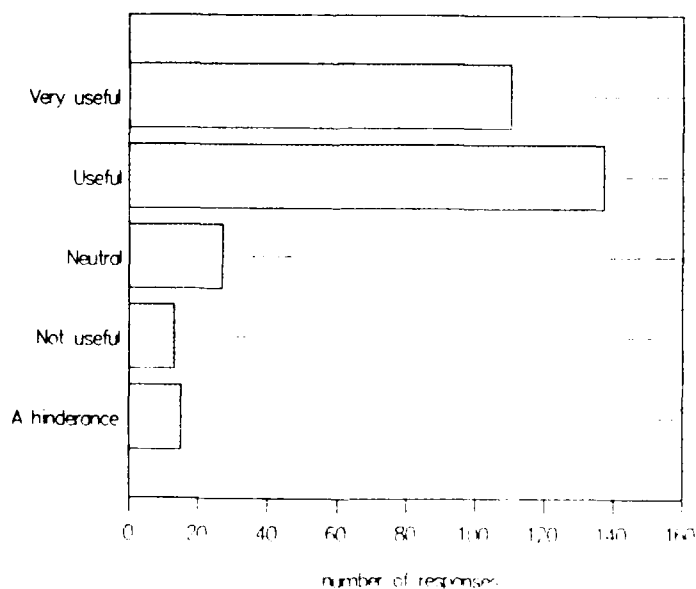
FIGURE 81. Chem 136, #4: In the role of designing experiments, acquiring data, and analyzing data, do you believe that computers are useful for learning chemistry in the laboratory?
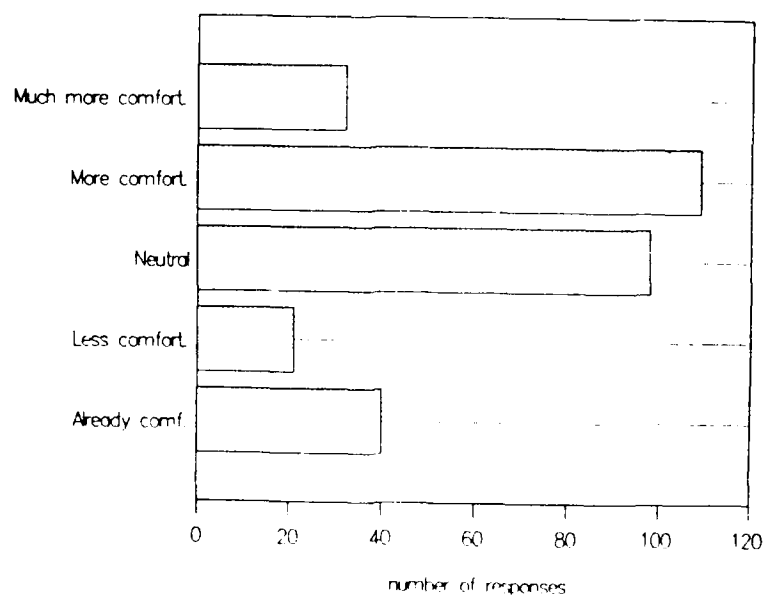
FIGURE 82. Chem 136, #5: Are you more comfortable with computers as a result of taking Chem 135 and Chem 136?
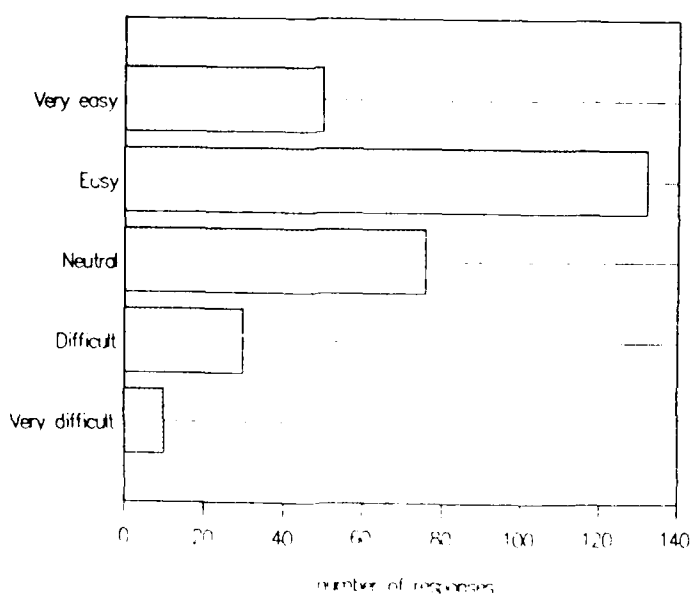
FIGURE 83. Chem 136, #6: Based on your experience in Chem 135 and Chem 136, were the laboratory computers easy to use?

Similar to Chem 125/135, most Chem 136 students (71%) gave an *easy* or

neutral response to the question about the readability and understandability of the lab

book (Figure 84). This gives us confidence that we are writing the laboratory

materials at a level and clarity representative of the students' background.
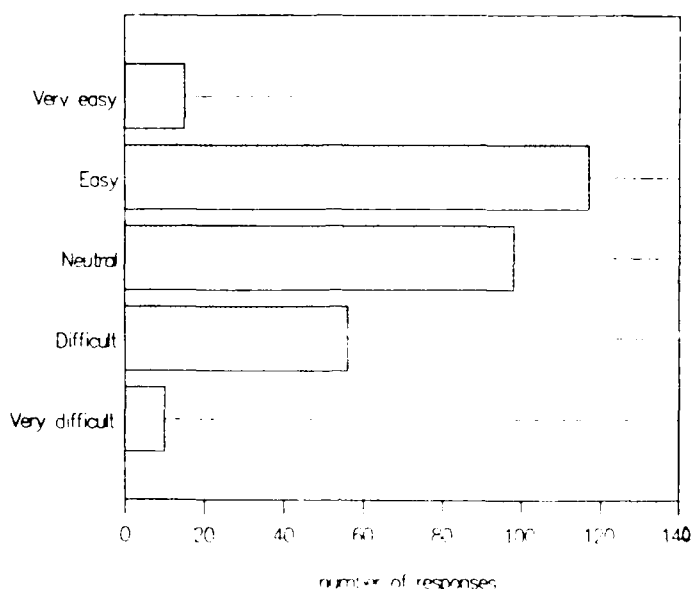
FIGURE 84. Chem 136, #7: Was the Chem 136 lab book easy to read
and understand?

In a general interpretation of the surveys, the students perceive that we are

providing a challenging, well organized course. Students are very supportive of the

final project concept. They overwhelmingly find the computers useful for learning
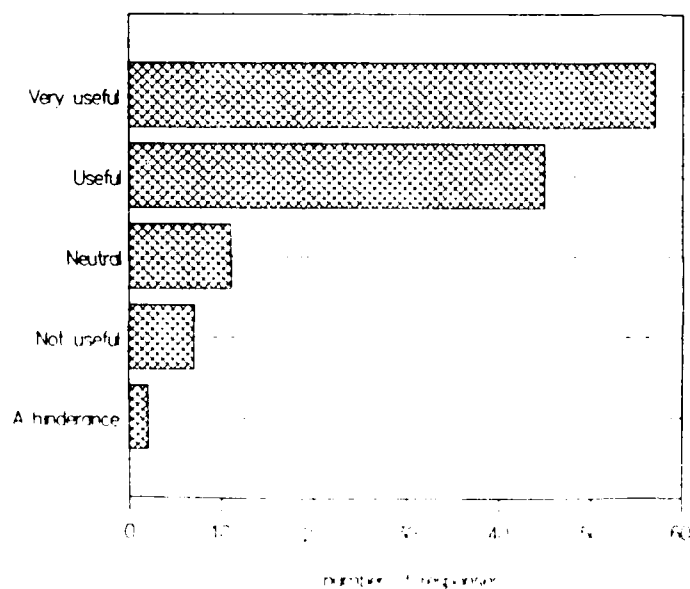
chemistry in the laboratory. We believe this support comes from the fact that we have

carefully integrated the process of science into our hardware and software. Students

find the laboratory computers easy to use and the majority are leaving the courses

more comfortable with computers. Irrespective of the computer's benefit in

chemistry, students are leaving our courses with a better feel for and appreciation of

the usefulness of computers. *Any* experience students receive with computers will

benefit them in their academic and vocational careers.

Students also gave favorable responses to the questions regarding the readability and understandability of the computer book and lab books. This is due in no small part to the preparation of the lab instructors. The summer training course made the lab instructors much more prepared to answer questions regarding the labs and the computers. The summer course gave the instructors the background and confidence to trouble shoot problems in the lab. which makes up for many shortcomings which may be present in the written materials Many of the written comments on the surveys were in support of the attitude and ability of the student's lab instructor.

Recommendations for Future Work

Our approach to using computers in the chemistry laboratory is unique in the country. A comparable integrated hardware/software system and set of curriculum materials simply does not exist elsewhere on any scale, large or small. The uniqueness of the system and relatively short time span in which it has been implemented implies that there is still a considerable amount of development work remaining to be done.

Perhaps the area in which the most additional work is needed is experiment design. Considerable time is needed to research, design, and test experiments which take advantage of the capabilities of the MSU lab interface system. This requires breaking a mind-set on the researcher's standpoint that the same labs must be done in general chemistry that have been done for the last 20 years! Certainly, the lab interface system is a useful instrument if it can do the same labs faster, easier. and more accurately than in a conventional laboratory. But the true utility of the system is when it is used to design and perform experiments that *cannot* be done in a conventional laboratory, allowing students to visualize chemical concepts which have not yet been explored in the general chemistry laboratory.

Coupled with the need for experiment design work is the need to develop new sensors. Any device that creates a current, voltage, or undergoes a resistance change can be used as a sensor. This includes interfacing *other* instruments (such as spectrophotometers and gas chromatographs) with the MSU lab interface. A cheap, reliable, easy-to-use pressure sensor is definitely needed. A variety of inexpensive ion-selective electrodes would also be very useful. With a trend toward microscale experiments, the MSU lab interface can be the ideal tool to use for quantitative measurements on small quantities of chemicals with additional sensor development.

The computer book and lab books associated with the MSU lab interface system are in constant need of revisions and updates. With student feedback and the development of new experiments, the format, style, and content of the written materials can only improve. Revisions to written materials for laboratory courses are often overlooked because the actual work is tedious and time consuming. Constructive revisions require not only the pen, but laboratory time as well. The less-than-glamorous task of revising course materials is extremely important and is a job that is never truly complete.

Incorporating computers into introductory science laboratories at the college level on a large scale is truly "revolutionary". Computers offer us a chance to teach laboratory science in the manner in which it is actually done. Through the MSU lab interface project, we have seized this opportunity! This has required developing new curriculum materials which take advantage of computers in the labs. This has required the "rethinking" of the types of labs we do, taking advantage of the new worlds of experimentation which microcomputer-based laboratories can open up to us.

Computers are no longer viewed as a novelty or as something "neat" to have in the laboratories at MSU; they are an integral part of the chemistry labs. There are many unique administrative, logistical, and technical issues associated with having laboratory computers on a large scale, and we have addressed many of these issues. As President Bush stated in his 1990 State of the Union address, America must regain its prominence in science education. The "re-tooling" of America's teaching laboratories is essential in meeting the President's goal. The microcomputer-based laboratory can prepare us for the 21st century.

153

REFERENCES CITED

REFERENCES CITED

1. Castleberry, S.; Lagowski, J.J. "Individualized Instruction Using Computer Techniques." J.Chem.Educ., 1970, 47(2), 91.

2. Hileman, B. "Computers in Undergraduate Chemistry Education." Chem. and Engr. News, 1988, 66(43), 29.

3. Feng, A.; Moore, J.W. "Exploring Chemistry by Computer: KC?Discoverer." J.Chem.Educ., 1986, 63(4), 327.

4. Harwood, W.S.; Moore, E.A.; Moore, J.W.; Susskind, T.Y. "The KC?Discoverer Workbook."; Project SERAPHIM: Eastern Michigan University, Ypsilanti, MI, 1987.

5. Furstenau, R.P.; Amend, J.R. "Applying KC?Discoverer in the Introductory Chemistry Laboratory." J.Chem.Educ., publication date, June 1990.

6. Weyh, J.; Crook, J.; Hauge, L. "Tools of Chemistry." Trinity Software, Inc., 1989.

7. Weyh, J. "Computers in General Chemistry: From Drill and Practice to Simulation." A seminar to the Department of Chemistry, Montana State University, Bozeman, MT, 1 February 1990.

8. Smith, S.G.; Chabay, R.; Kean, E. "Introduction to General Chemistry." Falcon Software, Inc., Wentworth, N.H., 1989.

9. Smith, S.G. "Computer-Assisted Instruction on a Microcomputer." J.Chem.Educ., 1984, 61(10), 864.

10. Swinnerton, J.W.; Miller, W.W. "Use of Digital Computer for a Solving Complex Chemical Equilibrium." J.Chem.Educ., 1959, 36(10), 485.

11. Bard, A.J.; King, D.M. "General Digital Computer Program for Chemical Equilibrium Calculations." J.Chem.Educ., 1965, 42(3), 127.

12. Emery, A.R. "Computer Program for the Calculation of Acid-Base Titration Curves." J.Chem.Educ., 1965, 42(3), 131.

13. Casanova, J. Jr.; Weaver, E.R. "Introduction to Computer Use: Program for First Order Rate Constants." J.Chem.Educ., 1965, 42(3), 137.

14. Boyko, E.R.; Whittaker, G.A. "Huckel Molecular Orbital Theory Calculations." J.Chem.Educ., 1965, 42(1), 48.

15. Cromer, D.T. "Stereo Plots of Hydrogen-like Electron Densities." J.Chem.Educ., 1968, 45(10), 626.

16. Davis, L.P.; Storch, D.M.; Strawser, L.D. "Physical Chemistry at USAFA: Personalized Instruction." J.Chem.Educ., 1987, 64(9), 784.

17. Anderson, K.P.; Snow, R.L. "A Relative Deviation, Least Squares Method of Data Treatment." J.Chem.Educ., 1967, 44(12), 756.

18. Smith, E.D.; Mathews, D.M. "Least Square Regression Lines: Calculations Assuming a Constant Percent Error." J.Chem.Educ., 1967, 44(12), 757.

19. McWilliam, I.G. "Programmable Calculators. Some Practical Aspects of Simulated Experiments." J.Chem.Educ., 1974, 51(7), 482.

20. Clark, G.J.; Kuemmerle, E.W.; Lieto, L.R. "Programmable Calculators: Uses in Freshman Chemistry Laboratories." J.Chem.Educ., 1975, 52(7), 423.

21. Murgich, J. "Huckel Calculations Using a Programmable Pocket Calculator.", J.Chem.Educ., 1977, 54(7), 421.

22. MacKenzie, I.S. "Issues and Methods in the Microcomputer-Based Lab." J.Comp.Math.Sci.Teach., 1988, 7(3), 12.

23. IBM Personal Science Laboratory: Functional Description and Technical Specifications, 1989 pamplet.

24. Pavia, D.L. "SQUALOR: The Design of a Laboratory Simulation." Acad.Comp., 1990, 4(5), 8.

25. Jones, L.L.; Karloski, J.L.; Smith, S.G. "A General Chemistry Learning Center: Using the Interactive Videodisc." Acad.Comp., 1987, 2(1), 36.

26. Willis, C.J. "Another Approach to Titration Curves." J.Chem.Educ., 1981, 58(8), 659.

27. Moore, J.W. "Achieving the Computer's Potential in the Chemistry Curriculum." Acad.Comp., 1987, 2(3), 18.

28. Whisnant, D.M. "Scientific Exploration with a Microcomputer: Simulations for Nonscientists." J.Chem.Educ., 1984, 61(7), 627.

29. Gilbert, D.D.; Mounts, T.T.,II; Frost, A.A. "Computer-Graphics Emulation of Chemical Instrumentation: Absorption Spectrophotometers." J.Chem.Educ., 1982, 59(8), 661.

30. Gable, R.W. "SPEC20: A Highly Interactive Simulation of a Manual Spectrophotometer." J.Chem.Educ., 1988, 65(1), 26.

31. Schatz, P.F. "Using Computers for Training Instrument Operators." Acad.Comp., 1987, 2(1), 38.

32. Howard, G.D.; DuBois, T.D. "A Microcomputer-Controlled T-60 NMR Emulator." J.Chem.Educ., 1986, 63(8), 771.

33. Brownawell, M.L.; Filippo, J.S.,Jr. "A Program for the Synthesis of Mass Spectral Isotopic Abundances." J.Chem.Educ., 1982, 59(8), 663.

34. Personal communication with Dr Loretta Jones, 27 January 1990.

35. Russell, A.A. "From Videotapes to Videodiscs: From Passive to Active Instruction." J.Chem.Educ., **1984**, *61*(10), 866.

36. Brooks, D.W.; Lyons, E.J.; Tipton, T.J. "Laboratory Simulations by Computer-Driven Laser Videodiscs." J.Chem.Educ., **1985**, *62*(6), 514.

37. Mendenhall, M.H. "Microcomputer Controlled Data Acquisition and the Interaction of Students with Laboratory Computers." Acad.Comp., **1989**, *3*(7), 20.

38. Powers, M.H. "Review of Two Commercial Interfacing Packages." J.Comp.Math.Sci.Teach., **1989**, *8*(3), 73.

39. Snyder, H.D. "Computer Instrumentation and the New Tools of Science." JCST, **1989/1990**, *19*(3), 171.

40. Woerner, J.J. "The Apple Microcomputer as a Laboratory Tool." J.Comp.Math.Sci.Teach., **1987/1988**, *7*(1,2), 34.

41. DeJong, M.L.; Layman, J.W. "Using the Apple II as a Laboratory Instrument." The Physics Teacher, **1984**, *22*(5), 291.

42. Layman, J.W.; DeJong, M.L.; Nelson, J.H. *Teacher Tutorial for AAPT Microcomputer Workshop on Laboratory Interfacing Experiments Using the Apple II Microcomputer Gameport*; AAPT: College Park, MD, 1987.

43. DeJong, M.L. *Apple II Applications*; Howard W. Sams & Co.: Indianapolis, IN, **1983**.

44. Li, C.; Zhuang, Q. "A Microcomputer-Based Temperature Probe." J.Chem.Educ., **1988**, *65*(4), 344.

45. St.Louis, R.V. "Audiofrequency Measurements for Heat Capacity Ratios Using Apple II." J.Chem.Educ., **1987**, *64*(9), 788.

46. Krause, D.C. "The Computer-Based Laboratory." J.Chem.Educ., **1988**, *65*(10), 875.

47. Powers, M.H. "Voltage Measurements with the Computer Gameport." J.Comp.Math.Sci.Teach., **1987/1988**, *7*(1,2), 68.

48. Westling, B.D.; Bahe, M.E. "Interface It Yourself." The Science Teacher, **1986**, *53*(8), 45.

49. Jesberg, R.O., Jr.; Dowden, E. "Microchip Measuring." The Science Teacher, **1986**, *53*(7), 34.

50. Kamin, G.D.; Dowden, E. "A Thermistor Interface." The Science Teacher, **1987**, *54*(6), 58.

51. Horst, K.E.; Dowden, E. "Collect Chem Data by Computer." The Science Teacher, **1986**, *53*(8), 38.

52. Farrell, K.; Dowden, E. "Tracking Temperatures With Your Computer." The Science Teacher, 1988, 55(2), 42.

53. Assetto, A.R.; Dowden, E. "Getting a Grip on Interfacing." The Science Teacher, 1988, 55(6), 65.

54. Sievers, D. "The Game Port Connection." The Science Teacher, 1986, 53(6), 49.

55. Connolly, W. "Upgrading Radiation Detectors with the Commodore." The Physics Teacher, 1987, 25(5), 323.

56. Moore, J.W.; Miles, P.; Rasmussen, M.; Hartman, K.; Barker, P. "Inexpensive Computerized Experiments." J.Chem.Educ., 1986, 63(4), 326.

57. Annino, R.; Driver, R.D. Scientific and Engineering Applications with Personal Computers; John Wiley & Sons: New York, 1986.

58. Priest, J.; Snider, J. "Undergraduate Computer-Interfacing Projects." The Physics Teacher, 1987, 25(5), 303.

59. Thomas, R.N.; Kuo, J.E. "Teaching Computer Interfacing in Instrumental Analysis." J.Chem.Educ., 1989, 66(11), 947.

60. Koval, C.A.; Rutkowski, C.J.; Cowan, J.P. "Circuit for Optimally Matching Instrumental Output Voltage Levels to the Input Voltage Levels on Analog-to-Digital Converters." Anal.Chem., 1985, 57(6), 1163.

61. Champ Universal Laboratory Interface, Merlan Scientific: System Description and Technical Specifications, 1988 pamphlet.

62. DeJong, M.L. "Apparatus Evaluation: The Versatile Laboratory Aid, VELA." The Physics Teacher, 1984, 22(5), 334.

63. Lambert, A. "VELA in Place of Ticker Tape." Phys.Educ., 1988, 23, 230.

64. Levkov, J.S. "The Use of Commercial Spreadsheet Programs in the Science Laboratory." J.Chem.Educ., 1987, 64(1), 31.

65. Joshsi, B.D. "Problem Solving in Physical Chemistry Using Electronic Spreadsheets." J.Chem.Educ., 1987, 64(9), 790.

66. Kari, R.; Dubreuil, Y. "Use of a Spreadsheet to Collect Large Quantities of Student Experimental Data." J.Comp.Math.Sci.Teach., 1987/1988, 7(1,2), 16.

67. Powers, M.H. "Commercial Interfacing Packages: Part II -- Software and Applications." J.Comp.Math.Sci.Teach., 1989, 8(4), 88.

68. IBM Personal Science Laboratory Workshop, Institute for Academic Technology, Research Triangle Park, North Carolina, 15-16 February 1990.

69. Mallow, J.V. Science Anxiety: Fear of Science and How to Overcome It; Van Nostrand Reinhold Company: New York, 1981.

70. Chang, R. Chemistry, 3rd ed.; Random House: New York, 1988.

71. Amend, J.R.; Tucker, K.A.; Furstenau, R.P. "Using Computers to Involve Students in the Process of Science." Acad.Comp., 1989, 4(3), 20.

72. Amend, J.R.; Briggs, R.D.; Furstenau, R.P.; Tucker, K.A.; Howald, R.A.; Ivey, B.E. "Laboratory Interfacing for Science Courses in Montana Schools: A Project at Montana State University." J.Comp.Math.Sci.Teach., 1989, 9(1), 95.

73. Amend, J.R.; Howald, R.A.; Ivey, B.E.; Tucker, K.A. "An Integrated Workstation for the Freshman Laboratory." J.Chem.Educ., publication date April 1990.

74. Amend, J.R.; Furstenau, R.P.; Tucker, K. "Student-Designed Experiments in General Chemistry Using Laboratory Interfacing." J.Chem.Educ., tentative publication date July 1990.

75. Amend, J.R.; Furstenau, R.P.; Tucker, K.A.; Ivey, B.E. Computer Applications in the Science Laboratory; Montana State University: Bozeman, MT, 1988.

76. Tucker, K.A. MSU Laboratory Interface Reference Manual; Montana State University: Bozeman, MT, 1989.

77. Nicklin, R.C. "The Computer as a Lab Partner." JCST, 1985, 15(1), 31.

78. Barnett, W.B. "User Interface Design for Analytical Instruments: Art or Science?" Anal.Chem., 1988, 60(20), 1169A.

79. Furstenau, R.P.; Tucker, K.A.; Amend, J.R. Using Computers in the General Chemistry Laboratory; Montana State University: Bozeman, MT, 1989.

80. Amend, J.R.; Furstenau, R.P.; Tucker, K.A.; Larsen, R. "Drawing Relationships From Experimental Data: Computers Can Change the Way We Teach Science." J.Comp.Math.Sci.Teach., submitted for publication April 1990.

81. Fuller, R.G. "Applications Software Transforms Science Teaching." JCST, 1987, 16(4), 239.

82. Salin, E.D. "Computer Interfacing for Chemists." J.Chem.Educ., 1982, 59(1), 53.

83. Lowry, B.; Thomas, H. "A Computer Interfacing Course Using the Commodore 64." J.Chem.Educ., 1986, 63(1), 70.

84. Volker, E.J.; Eldridge, D.C. "Computers in Chemistry." JCST, 1986, 15(6), 519.

85. Amend, J.R.; Howald, R.A.; Furstenau, R.P.; Armold, M.T. Experiments for the General Chemistry Laboratory; Montana State University: Bozeman, MT, 1989.

86. Amend, J.R.; Howald, R.A.; Furstenau, R.P. Experiments for the General Chemistry Laboratory II: Chemistry 136; Montana State University: Bozeman, MT, 1990.

87. Furstenau, R.P.; Tucker, K.A. Experiments to Accompany the MSU Laboratory Interface; Montana State University: Bozeman, MT, 1990.

88. Powers, M.H. "Resistance Probes in the Science Laboratory: Part I, The Thermistor Thermometer." J.Comp.Math.Sci.Teach., 1987, 7(2), 52.

89. Chau, F.T.; Chan, K.D. "A Computer-Based Temperature System." Computers Chem., 1988, *12*(4), 301.

90. Kennedy, S.E.; Sparberg, E.B.; Sterrett, F.S.K. *Chemical Quantitative Analysis: A New Approach*; New York: Harper & Row, 1972.

91. Kenner, C.T. *Analytical Separations and Determinations*; New York: The Macmillan Company, 1971.

92. *Standard Methods for the Examination of Water and Wastewater. 15th ed.*; Washington D.C.: American Public Health Association, 1980.

93. Alberty, R.A.; King, E.L. "Moving Boundary Systems Formed by Weak Electrolytes. Study of Cadmium Iodide Complexes." J.Am.Chem.Soc., 1951, *73*(1), 517.

94. Aylett, B.J. in *Comprehensive Inorganic Chemistry. Vol 3*; Oxford: Pergamon Press, Ltd., 1973.

95. Sneed, M.C.; Brasted, R.C. in *Comprehensive Inorganic Chemistry. Vol 4*; Princeton, N.J.: D. Van Nostrand Company, Inc., 1955.

96. Hill, J.O.; Magee, R.J. "Advanced Undergraduate Experiments in Thermoanalytical Chemistry." J.Chem.Educ., 1988, *65*(11), 1024.

97. Whitten, K.W.; Gailey, K.D.; Davis, R.E. *General Chemistry. 3rd ed.*; Philadelphia: W.B. Saunders Co., 1988.

98. Murphy, J. "Determination of Phosphoric Acid in Cola Beverages." J.Chem.Educ., 1983, *60*(4), 420.

99. Walton, H.F. "A Simple Recording Titrator." J.Chem.Educ., 1973, *50*(11), 795.

100. Wilkins, C.L.; Perone, S.P.; Klopfenstein, C.E.; Williams, R.C.; Jones, D.E. *Digital Electronics and Laboratory Computer Experiments*; New York: Plenum Press, 1975.

101. Fox, J.N.; Shaner, R.A. "Automated Computer-Interfaced pH Titrations." J.Chem.Educ., 1990, *67*(2), 163.

102. Chau, F.T. "A pH Measuring System Using Lotus Measure[TM]." Computers Chem., 1990, *14*(1), 69.

103. Olsen, E.D. *Modern Optical Methods of Analysis*; New York: McGraw-Hill, Inc., 1975.

104. Friedlander, G.; Kennedy, J.W.; Miller, J.M. *Nuclear and Radiochemistry. 2nd ed.*; New York: John Wiley & Sons, Inc., 1964.

105. Amend, J.R. "Investigations in Nuclear Chemistry: Radioactive Decay and Half Life." The Science Teacher, 1966, *33*(5), 31.

106. Broadbank, R.W.C.; Harding, R.D. in "Letters to the Editor." J.Chem.Educ., **1961**, *38*(6), 329.

107. Broadbank, R.W.C.; Harding, R.D.; Patel, S.J. in "Letters to the Editor." J.Chem.Educ., **1963**, *40*(6), 328.

APPENDICES

APPENDIX A

CHEM 125/135 PROJECTS

FINAL PROJECT #1
TURBIDIMETRIC DETERMINATION OF SULFATE IN WATER
Difficulty Factor: 1.15

Introduction/Objective

You will be determining the concentration of sulfate ion (SO_4^{2-}) in various water samples. To accomplish this, you will form a $BaSO_4$ stable, turbid suspension in the water sample. You will measure the degree of turbidity in the sample using the lab interface and colorimeter module.

Background

There are many ions which exist in natural waters. We've all heard of "hard" water, which contains high amounts of calcium (Ca^{2+}) and magnesium (Mg^{2+}) cations. Anions, such as SO_4^{2-}, CO_3^{2-}, and Cl^-, are also found in natural waters. These ions find their way into our water when rainwater reacts with common minerals in rocks and soil. Gypsum ($CaSO_4 \cdot 2H_2O$), calcite ($CaCO_3$), and dolomite ($MgCa(CO_3)_2$) are a few of these common minerals. All natural waters contain some ions. "Mineral" waters, which are currently popular among some health nuts and yuppies, simply have more of these dissolved minerals in them than "normal" waters. The type and quantity of dissolved minerals will give water a characteristic "taste". In this experiment, you will determine the concentration of one of these ions, sulfate, in a variety of water samples. To do this, you will use a colorimeter and apply a concept known as Beer's Law.

Beer's Law. Sulfate ion can be removed from water by a precipitation reaction with barium ion in acidic solution:

$$Ba^{2+}(aq) + SO_4^{2-}(aq) \text{-----> } BaSO_4(s)$$
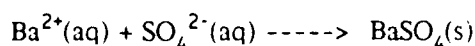
The acidic solution prevents the formation of unwanted precipitates. In theory, the white barium sulfate formed could then be filtered out of the solution and weighed to determine the sulfate concentration. In practice, however, the amount of barium sulfate formed is usually quite small, making it difficult to do such a measurement accurately.

A more accurate method involves suspending the fine particles of $BaSO_4$ in solution (a turbid suspension) and measuring the amount of light that passes through the solution. This is analogous to looking at the water at the edge of a lake. You can likely see the bottom of the lake bed at the edge of the lake if the water is calm. But if you disturb the water by agitating the bottom with a stick, you will likely see dirt and sand particles suspended in the water. These particles make it difficult to see the bottom. The more particles that are suspended (more concentrated), the more difficult it is to see the bottom.

Your colorimeter makes use of this same principle, as shown in Figure 1. As more $BaSO_4$ particles are in suspension (increase in turbidity), less light makes it through the sample to the detector. Turbidity is simply a property of a sample which causes light to be scattered and absorbed rather than transmitted in straight lines through the sample. The amount of light absorbed (absorbance, A) by the sample is directly proportional to the concentration (C) of suspended particles:

$$(1) \quad A = kC$$

where k is a constant. The value of k depends on the path length through the sample and the type of sample you're using (neither of which will change in your experiment). Essentially, the absorbance, A, is a measure of the turbidity of the sample. From Equation (1), a measurement of A will allow you to find the concentration.
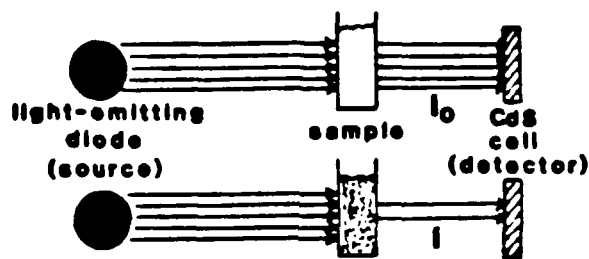
FIGURE 1. Principle behind Beer's Law

But wait a minute! Your colorimeter does not measure the amount of light absorbed by the sample! It measures the amount of light making it through the sample! This is known as the transmittance, or the fraction of light going through the sample. Since your detector measures current in relation to the amount of incident light, we will say that the current reading through a sample without a suspension is I_o. Without going through the details, the amount of light reaching the detector decreases exponentially as the light passes through the sample. If I represents the current reading through a sample with a suspension, the following relationship is true:

$$(2) \quad A = \log (I_o/I)$$

and $$(3) \quad \log (I_o/I) = kC$$

Equation (3) is known as a form of Beer's Law.

To find unknown sulfate concentrations, you must first construct a calibration curve based on measuring the transmittance of solutions of known sulfate concentration. The resulting plot of absorbance versus concentration should yield a straight line (if Beer's Law holds) which can be used to find unknown sulfate concentrations. The transmitted light is measured, the absorbance is then calculated, and the concentration of sulfate can be read off of your graph.

Experimental

Lab Interface Preliminaries

1. This project requires the use of a colorimeter. Your instructor will show you how to properly connect the colorimeter to the interface box.

2. The operation of the colorimeter is straightforward, as shown in Figure 2. The light-emitting diodes (LEDs) provide the light source. A CdS cell, described in Lessons 3, 4, and 5 of your Using Computers book, is the light detector.

FIGURE 2. Cut-away view of the colorimeter box.

3. For this experiment, you want to make sure that the **green** LED is connected to LED-A on the interface box. The command

SEND On TO LED-A

will turn "on" the green LED if it is properly connected.

4. You must also make sure that the CdS cell in the colorimeter operates properly. Refer to Lesson 3 in your Using Computers book.

Procedures

1. You will be given a bottle of water containing a known (100 mg/L) concentration of sulfate ion. You will want to make at least 5 different known concentrations (by dilution) from this standard sample. You will need at least 10.0 mL of each diluted sample which you prepare. The concentrations of your prepared dilutions should be relatively evenly spaced between and including 0 mg/L (distilled water) and the fully concentrated standard (100 mg/L). At this point, all of your solutions of known sulfate concentration will appear to be the same; this is because you have not yet made the barium sulfate suspension, which follows.

2. To obtain the barium sulfate suspension for each of your prepared standards, put 10 mL of each standard prepared in paragraph (1) into a clean test tube. Add 1 mL of 6 M HCl, 5 mL of 70% sorbitol solution, and 1.0 g of $BaCl_2 \cdot 2H_2O$ crystals. Measure all volumes as accurately as possible! *You will repeat the same preparation procedure for your unknown water samples, as well!*

3. After adding the reagents to the test tube, put a clean rubber stopper in the tube and shake vigorously. If the sample contains sulfate ion, a turbid suspension of barium sulfate should form. Let stand for a least 5 minutes. Then, shake the tube gently and pour a portion of the solution in the colorimeter cell and make a light transmission measurement using the green light-emitting diode. You will have to design the lab interface program which allows you to make this measurement.

4. This is not an easy experiment! It requires extremely good experimental technique; you must be very careful when preparing your standard solutions. If you don't feel you're getting good transmission readings with your standard samples, don't hesitate to prepare a new set of standards and try again! *To ensure accuracy, you must measure the transmission for your standards and your unknowns during the same lab period using the same colorimeter!*

Minimum Requirements

1. You must design an experiment which will send the photocell current readings and the concentrations of the <u>known</u> solutions to a data file. You must make the known solutions from a single standard provided by your lab instructor. You will include a listing of the program lines which accomplish this task with your final report.

2. You must use the **Spreadsheet** to get a plot of absorbance vs concentration for the <u>known</u> samples.

3. You will use the graph in (2) to find the sulfate concentrations of at least <u>three</u> unknown water samples. Your lab instructor will provide you with these unknowns, which may be prepared unknowns or bottled waters. If there are any particular water samples you would like analyze, you may do those samples instead of the ones provided by your instructor. Let your instructor know your plans!

4. You will submit <u>one</u> report (by you and your lab partner). Your lab instructor will inform you of the exact time and date the lab report is due.

5. You will give a short presentation (~5 minutes) about your project to your lab instructor and the rest of the class during the last lab period.

References

Roberts, J.L., Jr, Hollenberg, J.L., and Postma, J.M. <u>General Chemistry in the Laboratory</u>. New York: W.H. Freeman and Co., 1984.

Koniecko, E.S. <u>Handbook for Water Analysis</u>. Wayne, N.J.: Avery Publishing Group, Inc., 1982.

Sandell, E.B. and Onishi, H. <u>Photometric Determination of Trace Metals</u>. New York: John Wiley & Sons, 1978.

<u>Standard Methods for the Examination of Water and Wastewater</u>, 15th ed. Washington, D.C.: American Public Health Association, 1980.

Olsen, E.D. <u>Modern Optical Methods of Analysis</u>. New York: McGraw-Hill, Inc., 1975.

Verma, B.C., Swaminathan, K., and Sud, K.C. "An Improved Turbidimetric Procedure for the Determination of Sulphate in Plants and Soils." <u>Talanta</u>, **24**, 1977, 49-50.

FINAL PROJECT #2
THERMOMETRIC DETERMINATION OF CHLORIDE
CONCENTRATION IN WATER
Difficulty Factor: 1.10

Introduction/Objective

You will determine the concentration of chloride in two unknown solutions by thermometric titration with silver nitrate ($AgNO_3$).

Background

Chloride ion (Cl^-) is one of the major inorganic ions found in water and wastewater. In drinking water, the salty taste produced by chloride concentrations is variable and depends on the chemical contents of the water. Some water samples containing 250 mg chloride/liter may have a detectable salty taste if the cation is sodium. However, if the cations are calcium and magnesium, the salty taste may be absent in concentrations up to 1000 mg chloride/liter. High chloride content in water can be detrimental to metallic pipes and structural material, as well as plants.

Standard Methods details several methods for determining Cl^- concentrations in water. Several involve determining a color endpoint through titration. However, these methods require considerable solution preparation and considerable practice. The methods are quite accurate (better than 10 mg/L) in the 100-1000 mg chloride/L range.

If the chloride ion concentration is higher than 1000 mg/L, a much easier and faster method of determining chloride ion concentration is by a *thermometric titration*. Quite simply, the endpoint of the titration is determined by examining the changing slope of the temperature versus volume of titrant curve. For this titration, the chloride-containing sample is titrated with a known concentration of silver nitrate ($AgNO_3$). The following reaction occurs is the unknown chloride solution has the sodium cation:

$$(1)\quad AgNO_3(aq) + NaCl(aq) \longrightarrow AgCl(s) + NaNO_3$$

As the above reaction proceeds, a white precipitate forms. (You have seen the above reaction in your qualitative analysis lab.) In addition, the reaction is exothermic, so heat is evolved. As heat is evolved, the temperature of the reaction mixture will rise. When the reaction is complete, heat is no longer evolved and the temperature of the mixture will stabilize. The point at which the temperature stabilizes is the endpoint. It is important to note that the temperature rise associated with the amounts of solution you will use is quite small, but ideally suited for measurement by a thermistor and the lab interface.

Therefore, the *thermometric titration* of a chloride-containing sample with silver nitrate involves adding a known concentration of $AgNO_3$ to an unknown chloride solution. The curve will look similar to that shown in Figure 1.
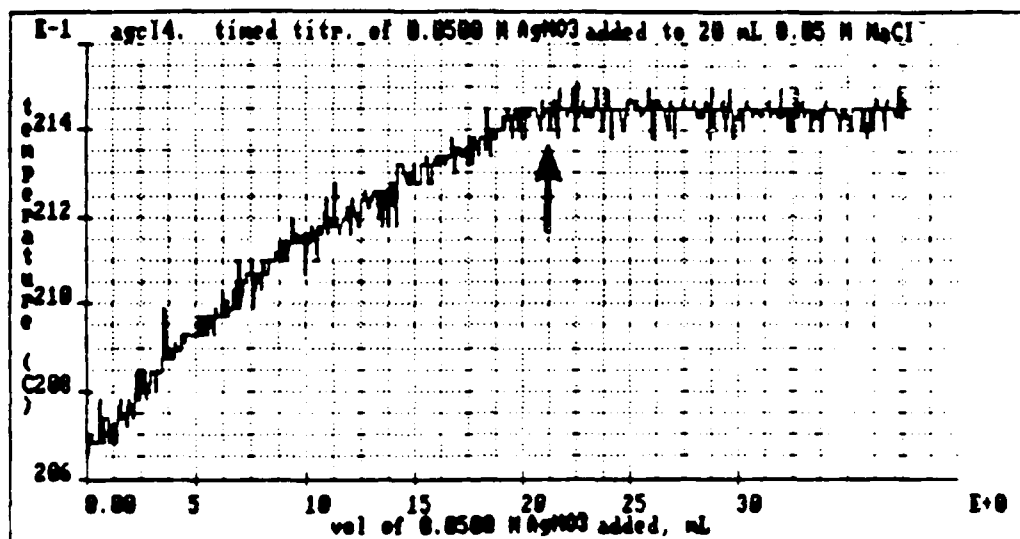
FIGURE 1. Thermometric titration curve.

At the thermometric endpoint (indicated by the arrow in Figure 1),

(1) moles of Ag^+ added $=$ moles of Cl^- in the unknown.

For molar concentration solutions,

(2) molarity X volume (in liters) = moles, so

(3) moles of Ag^+ added = (molarity) X (volume added).

Likewise for the chloride solution,

(4) moles of Cl^- in the unknown = (molarity) X (vol. of sample).

According to equation (1), the right sides of equations (3) and (4) can be set equal to each other. The titration allows you to find the volume of Ag^+ added. Thus, the only unknown is the molarity of the unknown chloride solution.

Experimental

Lab Interface Preliminaries

1. You will be using a thermistor to measure temperature. You should calibrate the thermistor using ice water and hot tap water.

2. You will need to design an experiment program which will send temperature and time (or volume) measurements to a **Spreadsheet** data file. You may also wish to actively graph the information on the screen while the experiment is running.

Procedures

1. Using a 500-mL volumetric flask, prepare a 0.05 M NaCl solution. Calculate the concentration of your solution to at least three significant figures. Pour about half of the solution into a clean, dry beaker and pour the remainder of the solution down the drain. This will be your "practice" solution.

2. Titrate 20.0 mL (measured with a pipette) of your "practice" NaCl solution with 0.05 M $AgNO_3$. *Note the exact concentration of the silver nitrate on the bottle!* The titration set-up is shown in Figure 2. You have two options in performing the titration:

a. *Perform a timed titration.* You can design a program that sends time and temperature to a data file every 1/2 second or so, with the buret dropping 2-3 drops per second. When the experiment is complete, you can use the **Spreadsheet** to convert time into volume, using a measured flow rate. To do this, however, you need a constantly flowing buret. This can be accomplished by first adjusting the flow of your buret to 2-3 drops per second, then pushing the titration beaker under the buret. Monitor the time it takes for the buret to flow 2-3 mL with the buret full. During the titration, keep adding $AgNO_3$ so that the buret level is in the same volume range as when you measured the flow rate. The flow rate (mL/sec) can then be multiplied by the time data using the **Spreadsheet** to obtain the volume of $AgNO_3$ added. This technique takes a little practice! It is important to continue stirring the solution throughout the titration.

b. *Perform a manual titration.* Basically, you will need to design a program that will do two things: send the total volume of $AgNO_3$ added to the chloride solution to a **Spreadsheet** data column, and (2) send the temperature value resulting from the latest addition of $AgNO_3$ to a second, corresponding **Spreadsheet** data column. A quick review of Lesson #5 from your Using Computers in the General Chemistry Laboratory book should give you the information you need to do this. The disadvantage of a manual titration is that it takes longer between measurements, increasing the likelihood of temperature instabilities.

3. Once you're convinced you can see the thermometric endpoint at the "expected" point for your practice solution, proceed to titrate two of the available unknown solutions. You must repeat each titration *at least* twice and take an average endpoint to determine the unknown chloride concentrations.
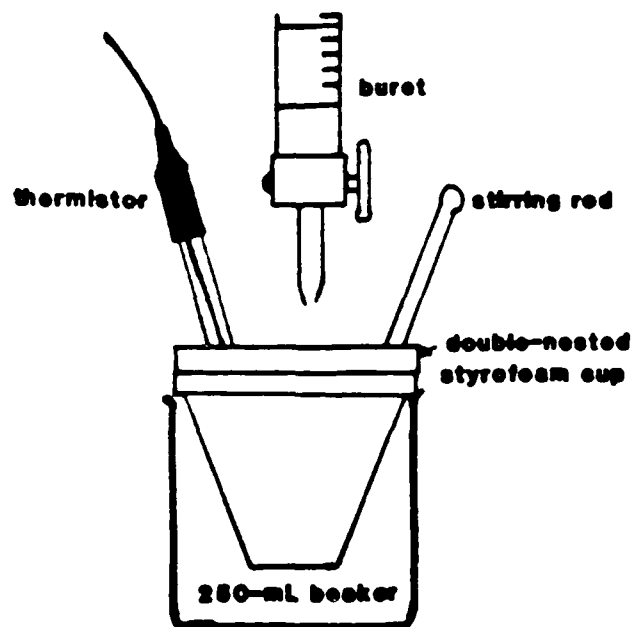
FIGURE 2. Set-up for thermometric titration.

Minimum Requirements

1. You must design an experiment which will send temperature and time (or volume) to a data file during the titration.

2. You must use the **Spreadsheet** to obtain temperature versus volume plots for each titration. From the plots, you will determine the endpoint and subsequently the chloride concentration of two of the unknowns.

3. Calculate the chloride ion concentration for each unknown in units of *molarity* and *mg chloride/liter of solution.*

4. You will submit one report (by you and your lab partner). Your lab instructor will inform you of the exact time and date the lab report is due.

5. You will give a short presentation (~5 minutes) about your project to your lab instructor and the rest of the class during the last lab period.

References

Chang, R. Chemistry, 3rd ed. New York: Random House, Inc., 1988.

Standard Methods for the Examination of Water and Wastewater, 15th ed., Washington D.C.: American Public Health Association, 1980.

FINAL PROJECT #3
DETERMINATION OF COPPER(II) IN AN UNKNOWN
Difficulty Factor: 1.05

Introduction/Objective

You will be determining the concentration of copper(II) ion in several unknowns. You will measure the concentration by light absorption using a colorimeter.

Background

Some of the principles involved in this project are closely related to those you learned in the spectroscopy lab. When electromagnetic radiation (light) interacts with matter, absorption of light will occur if the frequency of the light corresponds to the energy required to raise the matter to a higher allowed energy. A fundamental law regarding the amount of incoming light absorbed by a sample is **Beer's Law**.

If you take a full bottle of Coke and place beside it a bottle of Coke that is 1/10 Coke and 9/10 water, you will see a drastic difference in their appearance. To no great surprise, the full bottle of Coke will be a much darker brown color than the watered-down bottle. There are more of the molecules causing coloration in the full bottle of Coke than in the watered-down bottle. More of the visible light is being absorbed by the full bottle than the watered-down bottle. From this simple example, it seems reasonable that the amount of light absorbed by a sample (A) should be proportional to the amount (concentration, C) of absorbing species in the sample.

$$(1) \quad A = kC$$

This is the simplest form of Beer's Law. Beer's law is illustrated in Figure 1 on the next page.

FIGURE 1. Principle behind Beer's Law.

The k is a constant which depends on the path length through the sample (diameter of the Coke bottle) and the type of absorbing specie. As you will see, a measurement of the absorbance, A, of a sample will allow you to find the concentration of the light-absorbing specie. In other words, you can quantitatively identify chemical species in solution. Your colorimeter is an instrument designed to make these measurements.

Your colorimeter does not measure the amount of light absorbed by the sample. It measures the amount of light making it *through* the sample. The fraction of light going through the sample is known as the *transmittance*. In 1852, Beer discovered that the transmittance of light through a sample decreases exponentially (that is, logarithmically) as the concentration increases linearly. We will now put this in mathematical terms with respect to what the colorimeter can actually measure.

Your colorimeter contains a CdS cell which is exactly the same as you have used during your computer exercises. As you recall, the CdS light detector measures current in relation to the amount of incident light. Referring to Figure 1, we will say that the current reading for the cell without an absorbing specie in the path is I_o. If I represents the current reading through a sample with an absorbing specie, Beer's Law can be written mathematically as follows:

$$(2) \quad A = \log (I_o/I)$$

and
$$(3) \quad \log (I_o/I) = kC$$

To find unknown copper(II) concentrations, you must first construct a calibration curve based on measuring the transmittance of light through solutions of known copper(II) concentration. The resulting plot of absorbance versus concentration should yield a straight line (if Beer's Law holds), which can be used to find unknown copper(II) concentrations. The transmitted light is measured, the absorbance (log (I_o/I)) is then calculated, and the concentration of copper(II) can be read off of your graph.

There are a number of limitations to Beer's Law of which you should be aware. Beer's law is strictly valid at only low concentrations; thus, some deviations can be expected at higher concentrations. Beer's Law also assumes a monochromatic (single wavelength) light source is used. In reality, the green and red light emitting diodes (LEDs) of your colorimeter emit a band or range of wavelengths rather than a single wavelength. If the constant k in Equations (1) and (3) happens to vary in this band of wavelengths, Beer's Law will fail. The only limitation with which you will be concerned is concentration.

In this project, you will prepare your own "stock" solution of Cu^{2+}. You will then prepare a set of standard solutions from this stock solution via dilution. From your absorbance versus concentration graph of your standard solutions, you will determine the concentration range for which Beer's Law is valid. Using the linear region of your calibration graph, you will then determine the concentration of three unknown copper solutions.

Experimental

Lab Interface Preliminaries

1. This project requires the use of a colorimeter. Your instructor will show you how to properly connect the colorimeter to the interface box.

2. The operation of the colorimeter is straightforward, as shown in Figure 2. The light-emitting diodes (LEDs) provide the light source. A CdS cell, described in Lessons 3, 4, and 5 of your Using Computers book, is the light detector.

FIGURE 2. Cut-away view of the colorimeter box.

3. For this experiment, you want to make sure that the **red** LED is connected to LED-A on the interface box. The command

SEND On TO LED-A

will turn "on" the red LED if it is properly connected.

4. You must also make sure that the CdS cell in the colorimeter operates properly. Refer to Lesson 3 in your Using Computers book.

5. You will need to design an experiment program which will send light transmission current and concentration measurements to a **Spreadsheet** data file.

Procedures

1. You will be given a vial containing the solid form of a copper(II) compound. Using a 100-mL volumetric flask, prepare a 1 M solution of the compound containing the copper(II) ion. This 100 mL will be your "stock" solution of copper(II). You must calculate the exact molarity based on the formula of the compound and the actual amount you used.

2. You will want to make at least 8 different known concentrations from your "stock" solution by dilution. These known solutions should cover the range from the least concentrated possible (distilled water, 0 M) to the most concentrated possible (your "stock" solution, 1 M). You can prepare these known solutions by using the dilution equation:

$$M_s V_s = M_d V_d \; ,$$

where M_s represents the molarity of the starting material, V_s represents the volume of the starting material, M_d is the molarity of the diluted solution, and V_d is the volume of the diluted solution. The dilution equation is a conservation of moles equation. The number of moles of dissolved material stays the same when you dilute the solution; the molarity decreases and the volume increases. You should prepare at least 10 mL of each of your standards. This way, you can use a portion of one standard to prepare another of lesser concentration.

3. The more known samples you make, the better your calibration graph should be! You will make light transmission measurements using the **red** LED. You can use distilled water as the "blank"; that is, the transmission measurement for water (0 M) will be I_o.

4. Using the **Spreadsheet**, calculate the light absorbance for your known samples. Plot the absorbance versus the concentration. Identify the region of the graph which appears most linear (starting at 0 M) and make a linear regression plot of those points. It is this region of your absorbance/concentration calibration graph which will now be used to determine the concentration of the unknown copper(II) solutions.

5. You will now be given three samples of unknown copper(II) concentration. You will measure the light transmission through each sample and use your calibration graph to determine the concentration of each unknown. Some of the unknowns *may* have concentrations which are outside of the linear region of your calibration graph. If so, dilute the unknowns by a known factor until the absorbance is in the linear region of your calibration graph. By knowing the factor by which you diluted the solution, you can easily calculate the actual concentration of the unknown based on the concentration of the diluted unknown.

Minimum Requirements

1. You must design an experiment which will send the photocell readings and the concentrations of the known solutions to a data file. You must make the known solutions from a single 1 M standard which you prepare. You will include a listing of the program lines which accomplish this task with your final report.

2. You must use the **Spreadsheet** to get a plot of absorbance versus concentration for the known samples. You must determine the linear region of this plot and use this region to determine the concentration of your unknowns.

3. You will use the graph in (2) to find the copper(II) concentrations of three unknown samples. Your lab instructor will provide these unknowns.

4. You will submit one report (by you and your lab partner). Your lab instructor will inform you of the exact time and date the lab report is due.

5. You will give a short presentation (~5 minutes) about your project to your lab instructor and the rest of the class during the last lab period.

References

Olsen, E.D. Modern Optical Methods of Analysis. New York: McGraw-Hill, Inc., 1975.

FINAL PROJECT #4
DETERMINATION OF COBALT(II) IN AN UNKNOWN
Difficulty Factor: 1.02

Introduction/Objective

You will be determining the concentration of cobalt(II) ion in several unknowns. You will measure the concentration by light absorption using a colorimeter.

Background

Some of the principles involved in this project are closely related to those you learned in the spectroscopy lab. When electromagnetic radiation (light) interacts with matter, absorption of light will occur if the frequency of the light corresponds to the energy required to raise the matter to a higher allowed energy. A fundamental law regarding the amount of incoming light absorbed by a sample is **Beer's Law**.

If you take a full bottle of Coke and place beside it a bottle of Coke that is 1/10 Coke and 9/10 water, you will see a drastic difference in their appearance. To no great surprise, the full bottle of Coke will be a much darker brown color than the watered-down bottle. There are more of the molecules causing coloration in the full bottle of Coke than in the watered-down bottle. More of the visible light is being absorbed by the full bottle than the watered-down bottle. From this simple example, it seems reasonable that the amount of light absorbed by a sample (A) should be proportional to the amount (concentration, C) of absorbing species in the sample.

$$(1) \; A = kC$$

This is the simplest form of Beer's Law. Beer's law is illustrated in Figure 1 on the next page.

FIGURE 1. Principle behind Beer's Law.

The k is a constant which depends on the path length through the sample (diameter of the Coke bottle) and the type of absorbing specie. As you will see, a measurement of the absorbance, A, of a sample will allow you to find the concentration of the light-absorbing specie. In other words, you can quantitatively identify chemical species in solution. Your colorimeter is an instrument designed to make these measurements.

Your colorimeter does not measure the amount of light absorbed by the sample. It measures the amount of light making it *through* the sample. The fraction of light going through the sample is known as the *transmittance*. In 1852, Beer discovered that the transmittance of light through a sample decreases exponentially (that is, logarithmically) as the concentration increases linearly. We will now put this in mathematical terms with respect to what the colorimeter can actually measure.

Your colorimeter contains a CdS cell which is exactly the same as you have used during your computer exercises. As you recall, the CdS light detector measures current in relation to the amount of incident light. Referring to Figure 1, we will say that the current reading for the cell <u>without</u> an absorbing specie in the path is I_o. If I represents the current reading through a sample <u>with</u> an absorbing specie, Beer's Law can be written mathematically as follows:

$$(2) \; A = \log (I_o/I)$$

and
$$(3) \; \log (I_o/I) = kC$$

Equation (3) is also a form of Beer's Law.

To find unknown cobalt(II) concentrations, you must first construct a calibration curve based on measuring the transmittance of light through solutions of known cobalt(II) concentration. The resulting plot of absorbance versus concentration should yield a straight line (if Beer's Law holds), which can be used to find unknown cobalt(II) concentrations. The transmitted light is measured, the absorbance (log (I_o/I)) is then calculated, and the concentration of cobalt(II) can be read off of your graph.

There are a number of limitations to Beer's Law of which you should be aware. Beer's law is strictly valid at only low concentrations; thus, some deviations can be expected at higher concentrations. Beer's Law also assumes a monochromatic (single wavelength) light source is used. In reality, the green and red light emitting diodes (LEDs) of your colorimeter emit a band or range of wavelengths rather than a single wavelength. If the constant k in Equations (1) and (3) happens to vary in this band of wavelengths, Beer's Law will fail. What all these limitations mean is this: your data may not fit Beer's Law exactly, but it should be close!

In this project, you will prepare a set of standard solutions from a stock solution of Co^{2+} via dilution. From your absorbance versus concentration graph of your standard solutions, you will determine the concentration of three unknown cobalt(II) solutions.

Experimental

Lab Interface Preliminaries

1. This project requires the use of a colorimeter. Your instructor will show you how to properly connect the colorimeter to the interface box.

2. The operation of the colorimeter is straightforward, as shown in Figure 2. The light-emitting diodes (LEDs) provide the light source. A CdS cell, described in Lessons 3, 4 and 5 of your Using Computers book, is the light detector.

FIGURE 2. Cut-away view of the colorimeter box.

3. For this experiment, you want to make sure that the **green** LED is connected to LED-A on the interface box. The command

SEND On TO LED-A

will turn "on" the green LED if it is properly connected.

4. You must also make sure that the CdS cell in the colorimeter operates properly. Refer to lesson 3 in your Using Computers book.

5. You will need to design an experiment program which will send light transmission current and concentration measurements to a **Spreadsheet** data file.

Procedures

1. You will be given a bottle of known (0.30 M) concentration of cobalt(II) ion. You will want to make at least 5 different known concentrations (by dilution) from this sample. These known solutions should cover the range from the least concentrated possible (distilled water, 0 M) to the most concentrated possible (the stock solution, 0.30 M). You can prepare these known solutions by using the dilution equation:

$$M_s V_s = M_d V_d$$

where M_s represents the molarity of the starting material, V_s represents the volume of the starting material, M_d is the molarity of the diluted solution, and V_d is the volume of the diluted solution. The dilution equation is a conservation of moles equation. The number of moles of dissolved material stays the same when you dilute the solution; the molarity decreases and the volume increases. You should prepare at least 10 mL of each of your standards. This way, you can use a portion of one standard to prepare another of lesser concentration.

3. The more known samples you make, the better your calibration graph should be! You will make light transmission measurements using the **green** LED. You can use distilled water as the "blank"; that is, the transmission measurement for water (0 M) will be I_o.

4. Using the **Spreadsheet**, calculate the light absorbance for your known samples. Plot the absorbance versus the concentration of your known samples.

5. You will then be given three samples of unknown cobalt(II) concentration. You will measure the light transmission through each sample and use your calibration graph to determine the concentration of each unknown.

Minimum Requirements

1. You must design an experiment which will send the photocell readings and the concentrations of the known solutions to a data file. You must make the known solutions from a single standard provided by your lab instructor. You will include a listing of the program lines which accomplish this task with your final report.

2. You must use the **Spreadsheet** to get a plot of absorbance versus concentration for the known samples.

3. You will use the graph in (2) to find the cobalt(II) concentrations of three unknown samples. Your lab instructor will provide these unknowns.

4. You will submit one report (by you and your lab partner). Your lab instructor will inform you of the exact time and date the lab report is due.

5. You will give a short presentation (~5 minutes) about your project to your lab instructor and the rest of the class during the last lab period.

References

Olsen, E.D. Modern Optical Methods of Analysis. New York:
McGraw-Hill, Inc., 1975.

FINAL PROJECT #5
ELECTROCHEMISTRY
Difficulty Factor: 1.02

Introduction

You will be determining the voltage and current characteristics of batteries that you build.

Background

Electrochemistry is a branch of chemistry that affects you every day. Batteries which start your car, run calculators and wrist watches, and supply emergency power all operate on electrochemical principles. Corrosion of pipes, buildings, bridges, boats, and planes can be prevented with knowledge of basic electrochemical principles. Nerve impulses are the result of electrochemical reactions in our body.

At this point, it is very important that you get a good understanding of the basics of electrochemistry. Almost every good general chemistry text will have a chapter on electrochemistry. One text you may have easy access to is the book used by Chem 135-136 (Chemistry, 3rd ed., by Chang, 1988). Chapter 19 in Chang is devoted to electrochemistry. The library also has numerous general chemistry books which may be easier to understand than Chang.

You will be building **galvanic cells** (also known as **voltaic cells**). **Galvanic cells** act as batteries, taking advantage of the fact that substances can spontaneously lose or gain electrons. The result of the electrons being transferred between substances is electricity. One of the first commercial batteries, known as a Daniell cell, was developed in 1836. It looked similar to the figure on the next page.
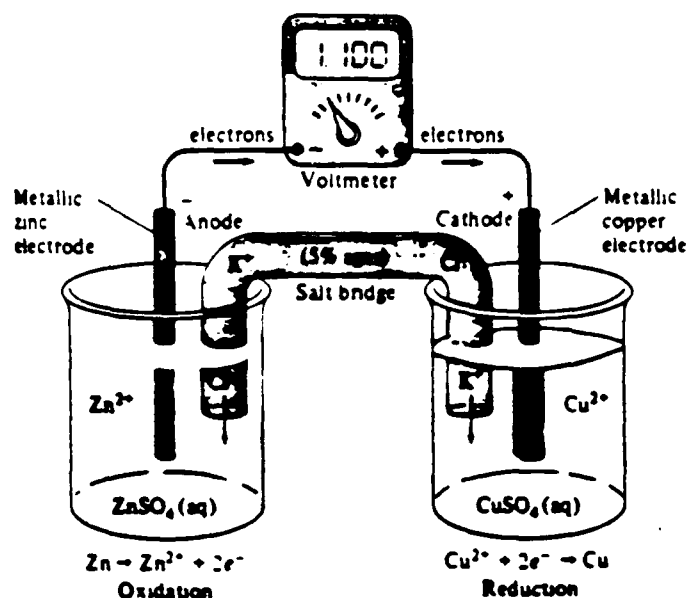
FIGURE 1. Daniell Cell (from Whitten, et.al.).

Although the above figure looks complicated, it is really fairly simple. Each compartment of the galvanic cell is known as a half-cell. The galvanic cell makes use of the fact that different substances (in this case, copper and zinc) have different tendencies to gain and lose electrons. The copper electrode wants to gain electrons more than the zinc does. Thus, the copper ions in the right half-cell will gain electrons from the zinc electrode. The copper ions are converted to neutral, metallic copper (reduction). Copper will plate out on the copper electrode as the reaction occurs. At the same time, the zinc electrode loses electrons and is converted to zinc ions (oxidation). Thus, the metal zinc electrode will eventually dissolve as it is converted into zinc ions. The result of these reactions is a flow of electrons (electric current) from the zinc electrode to the copper electrode . . . a battery! The salt bridge between the two half-cells allows electrical contact between the two solutions. The salt bridge also prevents mixing of electrode solutions and ensures electrically neutral solutions by providing a balancing flow of positive and negative ions. Theoretically, the above Daniel cell can provide 1.10 Volts. The exact voltage of a cell depends upon the materials used for the electrodes and the type/concentration of solution. Most chemistry texts with chapters on electrochemistry discuss how to calculate theoretical cell voltages.

To design an operational battery, you must choose some metal/solution/metal/solution combination that will provide the voltage you need. Sometimes, this requires hooking two or more batteries together, as is done in an automobile battery. Figure 2 shows two copper/zinc batteries hooked together in series. Notice the relative position of the electrodes and salt bridges. Notice also that the cathode of the left battery is connected to the anode of the right battery via a wire (a clip of some sort would also work). The relative position of the cells are important toward providing the proper polarity; that is, having the two copper electrodes hooked together would not work. Theoretically, the two copper/zinc batteries hooked together in series in Figure 2 should provide twice the voltage as one battery, or 2.20 Volts.
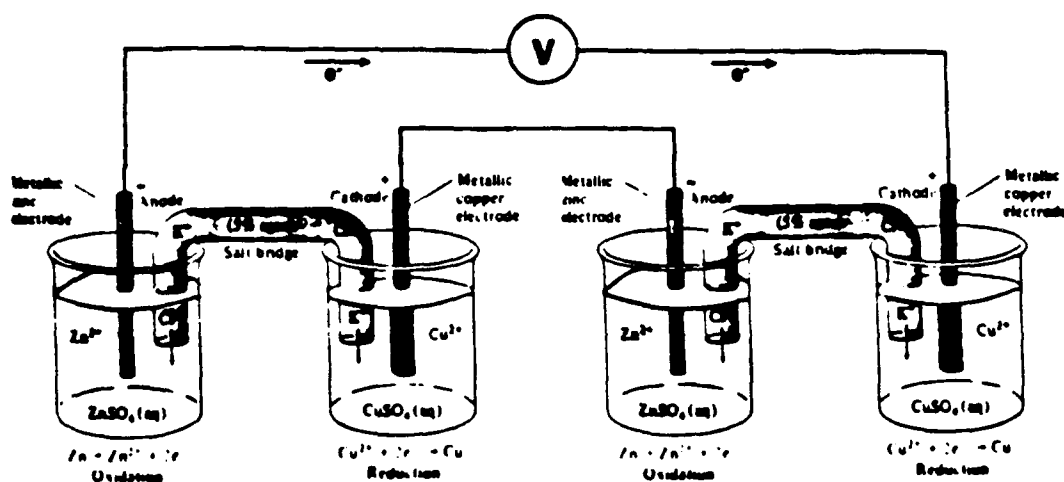
FIGURE 2. Two Daniell cells hooked in series.

In this project, you will test half-cell combinations to develop a battery which will provide at least 1.50 Volts under load conditions; that is, while lighting a light-emitting diode (LED).

Experimental

Lab Interface Preliminaries

1. For Part I, you must design an interface program which will allow you to use the interface as a simple voltmeter.

2. For Part II, you must design an experiment program which will send voltage and time information to a **Spreadsheet** data file for at least 5 minutes.

Procedures

Part I: Introduction

During the introduction to this project, you will experiment with various half-cell combinations to see which pairs can provide the voltage you need in Part II. Please read Part II before performing Part I, so you know the goals of the complete project before starting.

1. You will have the following metal/metal ion solution combinations available to you: $Ag/AgNO_3$; $Cu/Cu(NO_3)_2$; $Zn/Zn(NO_3)_2$; $Pb/Pb(NO_3)_2$. You will have two sets of metal electrodes.

2. You must test all six possible battery combinations of half-cells to determine the voltage from each combination. You will have to design an interface program to use as a voltmeter (this can be a simple modification to your temperature program from Lesson #2), measuring E1.

3. You will use 50 or 100 mL beakers to store the liquid for the half-cells. These beakers should be clean before using them.

4. Before setting up your batteries, polish the metal electrodes with steel wool to remove the oxide coatings.

5. For a salt bridge between half-cells, take one-half of a brown paper towel and roll it tightly like a cigar. Soak the rolled paper towel in a beaker of KNO_3 solution and remove it when you're ready to use it. You will need several of these salt bridges. Use a fresh salt bridge for each cell you build. You may handle the salt bridges with your fingers, but make sure you wash your hands at the end of the period. Throw used salt bridges in the garbage can, not into the sink!

6. To measure voltage properly, connect the red wire to the red post marked **E1**. Connect the black wire to the balck post below **E1**. Using the alligator clip ends of these wires, clip the wires onto the metal electrode in the half-cells. **Do not let the alligator clips touch the solution!** Figure 3 illustrates how this set-up will look for each half-cell combination:
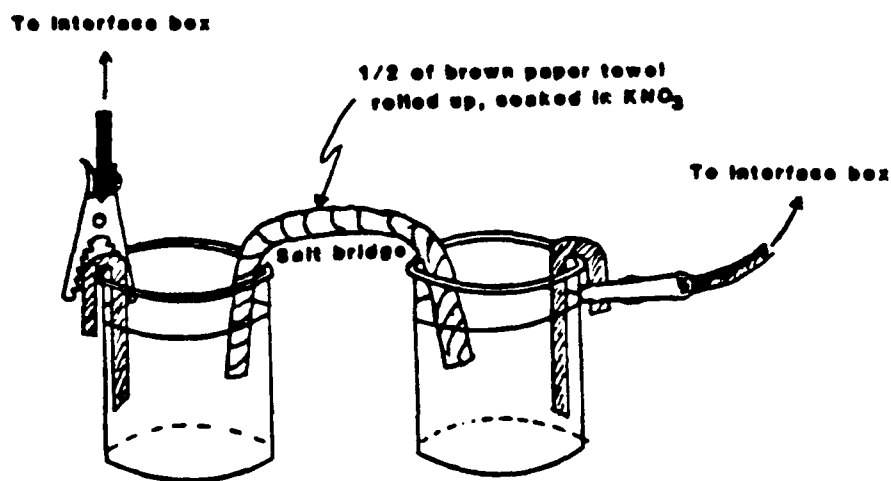
FIGURE 3. Galvanic cell configuration.
Note the two possible methods of
attaching the alligator clips to the
metal strip.

7. When measuring the voltage with your interface program, make sure you are getting a positive voltage. If you don't, simply reverse the position of the alligator clips on the metal electrodes. Note which color of wire is hooked to which electrode to give you the positive voltage. Note also that the voltage display from **E1** will read in millivolts. There is no need to send this voltage information for Part I to a **Spreadsheet** file; simply read the voltages from the monitor screen and write them down.

8. Compile your half-cell combination and voltage information into a neat table for your final report.

Part II: The Battery Design

1. Once you complete Part I, you are ready to design your battery. Based on your results from Part I,

Design a battery which will provide at least 1.5 Volts (1500 millivolts) for at least 5 minutes under load conditions; that is, it will have to light a light emitting diode (LED) while providing at least 1.5 Volts for 5 minutes.

2. You may use any combination of the cells you tested in Part I. **However, you may not use the same cell combination more than once.** For example, the configuration shown in Figure 2 is not acceptable, since Figure 2 shows two identical batteries hooked together. You may use one half-cell more than once, but not two identical complete cells.

3. To operate under load conditions, you must attach an LED to the E1 post as shown in Figure 3 below. Loosen the red and black colored plastic posts located at E1 to expose the holes in the metal posts.
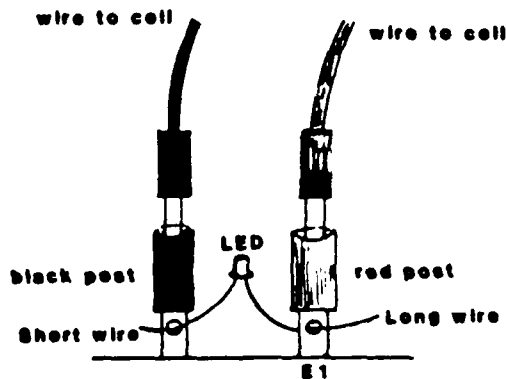
FIGURE 4. Side view of the E1 posts with the LED attached.

Insert the long LED wire into the hole in the red post and insert the short LED wire into the hole in the black post. Tighten the colored plastic portion of the posts until the LED is snug under the posts

4. To verify that your battery combination is performing up to requirements, you must send "voltage versus time" data to a **Spreadsheet** data file during the five minute "life" of your battery. A data point every one second is sufficient. This data will then be graphed using the **Spreadsheet** and turned in with your report.

5. Your lab instructor must also see the LED lit at the end of five minutes. Brightness is not important, just so the LED is visibly lit!

Minimum Requirements

1. You must include in your report a table summarizing the data from Part I. You do <u>not</u> need to include the program which allowed you to find voltage for Part I.

2. You must design an experiment(s) which will send voltage information with respect to time to a **Spreadsheet** data file for Part II. The program should take data every second. You will hand in a listing of the program which accomplishes these tasks.

3. You must include a sketch of your battery design from Part II.

4. You must include a plot of voltage versus time for your battery which spans at least 5 minutes (300 seconds).

5. You will submit <u>one</u> report (by you and your lab partner). Your lab instructor will let you know the exact time and date the report is due. Follow the format given on page 161 of your lab book.

6. On the last day of lab, you will give a short (~5 minutes) oral presentation about your final project to your lab instructor and the rest of the class.

References

Armold, M.T. and Amend, J.R. Experimental Chemistry. Montana
State University, 1988.

Amend, J.R. Chemistry. Montana State University, 1988.

Chang, R. Chemistry, 3rd ed. New York: Random House, Inc.,
1988.

Novicki, S. "Electrochemistry Applications Lab", U.S. Air Force
Academy, Spring 1988.

Whitten, K.W., Gailey, K.D., and Davis, R.E. General Chemistry,
3rd ed., Philadelphia: W.B. Saunders Co., 1988.

FINAL PROJECT #6
ACID/BASE PROPERTIES OF OVER-THE-COUNTER MEDICINES
Difficulty Factor: 1.0⁻

Introduction/Objective

You will determine the "acid-absorbing" properties of two common antacid products. You will also examine the buffering capacity of aspirin products. You will be using acid-base titration techniques to arrive at your results.

Background

Part I: Antacids

Before you begin this project, it is very important that you have a very good understanding of acids, bases, and titrations. Only some basics will be covered in this handout. If you are in Chem 125, you should understand Chapter 14, Acids and Bases, in your textbook by Amend. If you are in Chem 135, you should understand Section 16.8 in the textbook by Chang.

Commercial antacid tablets contain bases. People self-prescribe antacid tablets to relieve "heartburn" and "acid indigestion" which they feel is caused by the stomach excreting excess stomach acid (HCl). In principle, the base in the antacid neutralizes the excess stomach acid.

There are standard pharmaceutical testing techniques for evaluating antacid tablets. These techniques not only test for acid neutralizing capability, but account for gastric residence time, body temperature, the rate at which the antacid reacts, and the actual pH rise in the stomach. All of these factors are important physiologically. For example, if the antacid causes the pH of the stomach to rise above 3 (the stomach is normally around pH 1), the digestive enzyme pepsin begins to deactivate, causing digestion problems. In this project, you will only be concerned with determining the acid-absorbing capability of antacids.

Titration is a method which can be used to find the acid-absorbing capability of an antacid tablet. Titration techniques are commonly used to determine unknown concentrations of acids and bases. If the base concentration is unknown, a known amount of the base is titrated with a measured amount of a known concentration of acid. If the acid concentration is unknown (as shown in Figure 1 on the next page), the unknown is then titrated with a measured amount of a known concentration of base.
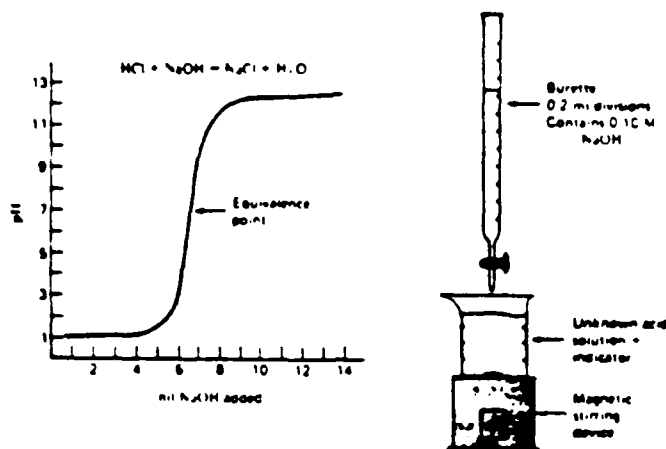
FIGURE 1. Titration of a strong acid with a strong base.

When monitoring the change in pH as base is added, the resulting solution will experience a rapid rise in pH at the equivalence point. At the equivalence point,

moles of acid = moles of base added

The middle of the steep rise (where the slope is the steepest) is the equivalence point. The moles of base can be found as follows:

moles of base added = (liters of base added)x(molarity of base)

It would seem that in order to find the acid neutralizing capability of an antacid, you could dissolve the tablet in water and titrate the solution with a known concentration of acid, such as HCl. However, many antacids are not very soluble in water. Therefore, you will dissolve the antacids in a known, excess amount of HCl. You will then titrate this resulting acidic solution with a known concentration of NaOH. Thus, at the equivalence point:

	moles of HCl =	moles of HCl	+	moles of HCl
Eqn. 1	added to	neutralized		neutralized
	antacid tablet	by antacid		by NaOH

We'll now refer to the above relationship as **Equation (1)**, where the moles of HCl and NaOH are found by multiplying their molarities (M) by the volume (V) used, in liters.

Thus, you will need "pH vs volume of NaOH added" titration curves for each antacid used in order to determine the equivalence point for each titration.

Part II: Buffering Capacity of Aspirin

You have all seen commercials touting the advantages of using one brand of aspirin over another. Some manufacturers say that buffered aspirin is better for you because it prevents stomach upset. If an aspirin is effectively buffered, the pH will not vary much upon the addition of moderate quantities of acid and base. You will measure how much acid or base it takes to make the pH of a solution of the aspirin change by one pH unit (an approximation of exceeding the buffering capacity).

Experimental

Lab Interface Preliminaries

1. For Part I of the project, you must design an experiment program which will send pH and volume (or time) information to a **Spreadsheet** data file during the titration. If you choose to do a *timed titration* (see *Procedures*), you must convert the time into a volume using the **Spreadsheet.**

2. For Part II, you must design an experiment program that simply displays a pH reading on the screen. You do not have to store the data on disk.

Procedures

Part I: Titration of Antacid Tablets

Preparation of the Antacid Sample. You will analyze <u>two different brands</u> of antacid tablets. For each tablet, use the following procedure.

1. Weigh an antacid tablet. Grind up the antacid tablet in a mortar and transfer the powdered sample to a clean 250 mL beaker. Rinse the mortar with a few milliliters of water to capture the remaining powder and add this water to the beaker.

2. Using a pipette or buret, put 50.0 mL of 0.3 M HCl into the beaker with the tablet (*note the exact concentration of the HCl on the bottle*). Heat the beaker to boiling with a hot plate, gently stirring the solution. Let the solution boil for 5 minutes, then let cool to room temperature. **SAFETY NOTE: Use extreme caution around the boiling acid!** You may hold the beaker under tap water to speed up the cooling (don't let water get into the beaker!).

Titrating the antacid tablets.

1. When the beaker has cooled, place a calibrated pH electrode in the beaker and use your pH titration program to measure the pH. This will be your first data point, with the total volume of NaOH added equal to zero. The pH should be acidic at this point.

2. Take data for pH versus the total volume of 0.3 M NaOH added, using a buret to add the 0.3 M NaOH (note the exact concentration of NaOH on the bottle). You should gently stir the solution each time you add NaOH and before you actually make your pH reading. This data should be sent to a data file for later graphing using the **Spreadsheet**. Small (0.1 mL or less) quantities of NaOH should be added when the pH begins to change significantly. This will ensure that you get several data points around the equivalence point and thus a more accurate graph.

3. Take data for a total of 5-10 mL of NaOH beyond the equivalence point. Graphing your data with the **Spreadsheet** will then allow you to easily find the equivalence point from the graph. Taking the derivative of the pH-volume curve will make the equivalence point even easier to find.

Note: You may also do a *timed titration*, where you're measuring pH versus time, once every 1/2 second or so, for a constantly flowing buret. However, you must convert time into a volume of NaOH to find the equivalence point. This procedure is much faster, but the program for the interface is more difficult.

4. Using **Equation (1)**, calculate the moles of base in the antacid tablet. Then, using the balanced equation for each active ingredient's (base) reaction with HCl, calculate the mass of active ingredient in the tablet. Refer to the end of this project description for a table of these reactions. Compare this with the value given on the manufacturer's label. Also, find the mass percent of inert ingredients in each tablet. Organize your data/calculations so they're easy to follow!

Note: If you choose Digel, you will notice that it uses two active ingredients: $CaCO_3$ and $Mg(OH)_2$. In your calculations, assume that the *mole ratio* of $CaCO_3$ to $Mg(OH)_2$ in the tablet is 1.27-to-1.

Part II: Buffering Capacity of Aspirin

1. Dissolve one tablet of each aspirin product (one plain aspirin, one buffered aspirin) in 100 mL of water. Each tablet will be in a separate solution! With a calibrated pH electrode in the solution, slowly (drop-by-drop) add 1 M HCl to the plain aspirin solution, monitoring the change in pH. When the pH is one unit different from when you started, note the total volume of HCl added (in drops). This is the amount of acid needed to exceed the buffering capacity of the aspirin. You do not need to send any of this data to the **Spreadsheet** or plot it! You may simply use the pH program you built for Experiment 7.

2. Repeat this procedure for the buffered aspirin solution and for 100 mL of water without an aspirin product dissolved. The water sample will be your standard for comparison.

3. Once the addition of HCl is completed, pour the solutions down the drain and prepare new 100 mL water solutions of each aspirin product and one beaker of 100 mL of plain water. Now, monitor the change in pH with the addition of 1 M NaOH. This will give you an idea of the base buffering capability of each product.

4. Construct a table which summarizes your results for this part of the experiment and comment on the results.

Minimum Requirements

1. You must design an experiment which will send pH readings and total volumes of NaOH added to a data file for Part I. You will hand in a listing of the program lines which perform the task.

2. You must use the **Spreadsheet** to get a plot of pH versus mL of NaOH added for each of the two brands of antacid tablets. You will use these plots to find the equivalence point of each titration.

3. You will use the titration results to perform all calculations necessary to find the grams of active ingredient in each antacid tablet and the weight percent of inert ingredients.

4. You will hand in a table of results for Part II and comment on the results.

5. You will submit <u>one</u> report (by you and your lab partner). Your lab instructor will inform you of the exact time and date the lab report is due. Follow the format given on p 161 of your lab book.

6.   ⅃ will give a short presentation (~5 minutes) about your project to your lab instructor and the rest of the class during the last lab period.

References

<u>USAF Academy Chemistry Demonstration Manual</u>, 10th ed., 1987.

Roberts, J.L., Jr, Hollenberg, J.L., and Postma, J.M. <u>General Chemistry in the Laboratory</u>. New York: W.H. Freeman and Co., 1984.

Chang, R. <u>Chemistry</u>, 3rd. ed. New York: Random House, 1988.

Hem, S.L. "Physicochemical Properties of Antacids," <u>J.Chem.Educ.</u>, 52 (6), 1975, 383-385.

Batson, W.B. and Laswick, P.H. "Pepsin and Antacid Therapy: A Dilema," <u>J.Chem.Educ.</u>, 56 (7), 1979, 484-486.

<u>Appendix: Reactions of Antacid Tablets</u> (from Chang, p 634)

$$NaHCO_3 + HCl \longrightarrow NaCl + H_2O + CO_2$$

$$CaCO_3 + 2HCl \longrightarrow CaCl_2 + H_2O + CO_2$$

$$MgCO_3 + 2HCl \longrightarrow MgCl_2 + H_2O + CO_2$$

$$Mg(OH)_2 + 2HCl \longrightarrow MgCl_2 + H_2O$$

$$Al(OH)_2NaCO_3 + 4HCl \longrightarrow AlCl_3 + NaCl + 3H_2O + CO_2$$
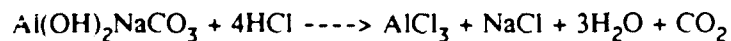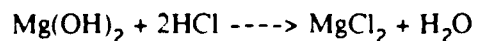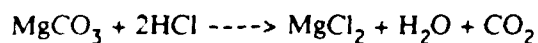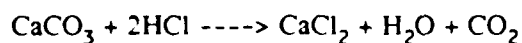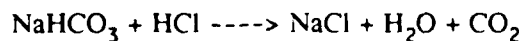
FINAL PROJECT #7
ACID/BASE PROPERTIES OF COMMON HOUSEHOLD PRODUCTS AND FOOD
Difficulty Factor: 1.00

Introduction/Objective

You will be determining the acid/base properties on many common household and food products. You will also be using acid/base titration techniques to determine the percentage of hydrochloric acid in a common liquid toilet bowl cleaner.

Background

Before you begin this project, it is very important that you have a very good understanding of acids, bases, pH, and titrations. Only some basics will be covered in this handout. If you are in Chem 125, you should understand Chapter 14, Acids and Bases, in your textbook by Amend. If you are in Chem 135, you should review Chapter 16 in the textbook by Chang.

Acids and bases are chemical compounds which we encounter constantly in our everyday lives. It is very often the acid/base properties of a food product which give it a characteristic taste. (CAUTION! DO NOT TASTE ANY OF THE CHEMICALS USED IN THIS LAB!) It is also the acid/base properties of many household products which cause them to work.

In this project, you will measure the pH of many food and household products. You will determine what ingredient is responsible for the pH and what (if any) function this ingredient has in the product. In addition, you will quantitatively measure the percentage of hydrochloric acid in a popular liquid toilet bowl cleaner.

Experimental

Lab Interface Preliminaries

1. For Part I of the project, you must design an experiment program that simply displays a pH reading on the screen. You do not have to store this data on a disk.

2. For Part II, you must design an experiment program which will send pH and volume (or time) information to a **Spreadsheet** data file during the titration. If you choose to do a *timed titration* (see *Procedures*), you must convert the time into a volume using the **Spreadsheet**.

Procedures

CAUTION! DO <u>NOT</u> MIX ANY OF THE HOUSEHOLD PRODUCTS TOGETHER! VERY TOXIC GASES MAY RESULT!

Part I: pH of Food and Household Products

To better organize your data, set up a data table which includes <u>at least</u> the following: product/name brand, diluted/straight, pH, color, active ingredient, and function. For some of the household products, you may want to include chemical equations which describe how the active ingredient works (this may require some outside research).

1. For all food (consumable) products, you may take the pH of the straight product (e.g. orange juice, Coke).

2. For all liquid household products, dilute 1 volume of product to 5 volumes solution with distilled water. This dilution will make the products safer to handle.

3. For solid household products, add 50 mL of water per gram of solid (e.g. detergents, crystal bowl cleaners).

4. Measure the pH of all of the available products using the lab interface. You may use the program you developed for Experiment 7 to measure pH for this portion of the project. You may test products of your own, if you wish.

Part II: Quantitative Measurement of HCl in a Liquid Toilet Bowl Cleaner

In this part of your project, you will find the percent hydrochloric acid in Lysol Liquid Toilet Bowl Cleaner (CAUTION! AVOID CONTACT WITH SKIN! If contact occurs, rinse promptly under the sink and get your lab instructor!) Hydrochloric acid (HCl) is the active ingredient in many liquid toilet bowl cleaners. HCl will react with hard water scale (calcium carbonate) in an acid/base reaction which dissolves the scale. Because of the concentrated acidic nature of the bowl cleaner, it also acts as a disinfectant. Other ingredients are often added to enhance the germicidal properties and flow characteristics.

Titration is a method which can be used to find the amount of HCl in the bowl cleaner. Titration techniques are commonly used to determine unknown concentrations of acids and bases. If the acid is the unknown, the unknown is titrated with a measured amount of a known concentration of base.
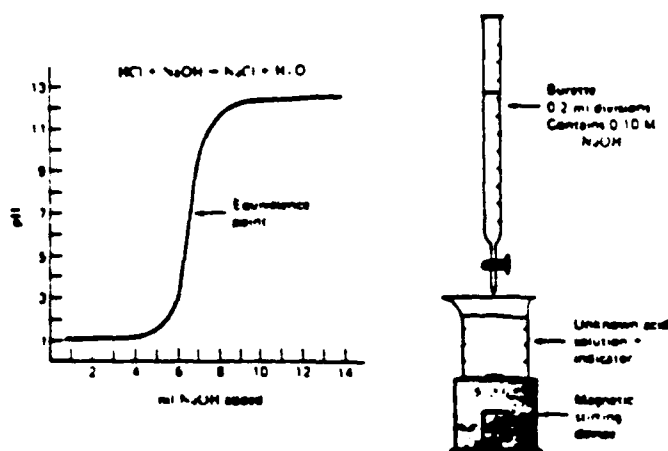
FIGURE 1. Titration of a strong acid with a strong base.

When monitoring the change in pH as base is added, the resulting solution will experience a rapid rise in pH at the equivalence point. At the equivalence point,

moles of acid = moles of base added

The middle of the steep rise (where the slope is the steepest) is the equivalence point. The moles of base added can be found as follows:

moles of base added = (liters of base added)x(molarity of base)

Thus, you will need a "pH versus volume of NaOH added" titration curve to find the volume of base added at the equivalence point. You will need to design an experiment for the lab interface which allows you to obtain a pH vs volume graph.

1. To prepare the bowl cleaner for titration, add 5.0 mL of liquid bowl cleaner (using a pipette or graduated cylinder) to a dry, clean, previously weighed 250 mL beaker. Weigh the beaker and bowl cleaner to obtain the mass of the bowl cleaner. After you've determined the mass of the bowl cleaner, add about 40 mL of distilled water.

2. Place a calibrated pH electrode in the beaker and use the pH titration program which you've designed to measure the pH of the solution. Make sure the glass bulb of the pH electrode is totally immersed in the solution. This will be your first data point, with the total volume of 0.5 M NaOH added equal to zero (note the exact concentration of NaOH on the bottle). Take data for pH versus the total volume of NaOH added. You should gently stir the solution each time you add NaOH and before you actually make your pH reading. This data should be sent to a data file for later graphing using the Spreadsheet. Small (0.1 mL or less) quantities of NaOH should be added when the pH begins to change significantly.

This will ensure that you get several data points around the equivalence point and thus a more accurate graph.

3. Take data for a total of 5 to 10 mL of NaOH beyond the equivalence point. Graphing your data with the **Spreadsheet** will then allow you to easily find the equivalence point from the graph.

Note: You may also do a timed titration, where you're measuring pH versus time, once every 1/2 second or so, for a constantly flowing buret. However, you must convert time into a volume of NaOH to find the equivalence point. This procedure is <u>much</u> faster experimentally, but the program for the interface is more difficult to write.

4. From the graphically-obtained equivalence point, find the moles of HCl in the 5.0 mL sample of toilet bowl cleaner. Then, calculate the grams of HCl in the sample. Knowing the mass of bowl cleaner you started with, calculate the mass percent of HCl in the 5.0 mL sample. How does this compare with what the manufacturer says on the label? Organize your calculations so they're easy to follow! You should repeat the titration as many times as possible and average the results.

Minimum Requirements

1. You must turn in a table of your results from Part I.

2. You must design an experiment which will send pH readings and total volumes of NaOH added to a data file for the liquid bowl cleaner titration. You will hand in a listing of the program lines which perform the task.

3. You must use the **Spreadsheet** to get a plot of pH versus mL of NaOH added for the liquid bowl cleaner. You will use the plot to find the equivalence point. You may want to make more than one titration and average your final results for mass percent.

4. You will use the titration results to perform all calculations necessary to find the mass percent of hydrochloric acid in the liquid bowl cleaner.

5. You will submit <u>one</u> report (by you and your lab partner). Your lab instructor will inform you of the exact time and date the lab report is due. Follow the format given on p 161 of your lab book.

6. You will give a short presentation (~5 minutes) about your project to your lab instructor and the rest of the class during the last lab period.

References

Amend, J.R. <u>Chemistry</u>. Montana State University, 1988.

APPENDIX B

CHEM 136 PROJECTS

CHEM 136
FINAL PROJECT #1
KINETICS OF THE BROMINE/FORMIC ACID REACTION
Difficulty Factor: 1.05

Introduction/Objective

You will be studying the kinetics of the bromine/formic acid reaction colorimetrically. As the reaction proceeds, the color intensity of the bromine decreases, making it possible to measure this decrease with the block colorimeter.

Background

The reaction between molecular bromine (Br_2) and formic acid (HCOOH) is discussed quite well in your textbook by Chang, beginning on page 525. In fact, it would be very beneficial to read this discussion before attempting this laboratory. The reaction proceeds as follows:

(1) Br_2(aq) + HCOOH(aq) -----> $2Br^-$(aq) + $2H^+$(aq) + CO_2(g)
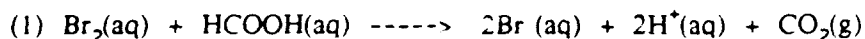
Molecular bromine has a characteristic brown-orange color in aqueous solution. As the above reaction proceeds, the color of the Br_2(aq) fades as it is converted to colorless bromine ion (see Figure 13.3 on page 525 in Chang). Therefore, the progress of the reaction can be monitored with a colorimeter.

As you have already seen in experiment #2, Beer's Law can be used to find concentrations of colored species using the colorimeter. The amount of light absorbed by a sample (A) is directly proportional to the concentration of the absorbing specie:

(2) $A = kC$

Remember, too, that the colorimeter does not directly measure the amount of absorbed light; it measures the amount of *transmitted* light. If I represents the transmission current for the absorbing specie and I_o represents the transmission current for a sample *without* the absorbing species (a blank), the absorbance can be expressed as follows:

(3) $A = \log (I_o/I)$

and   (4) $\log (I_o/I) = kC$

For the absorbing specie (Br_2) in this project, k is equal to 2.695 M^{-1}. Therefore, you can calculate the concentration of Br_2 simply by measuring the transmission currents and determining I_o.

Experimental

Lab Interface Preliminaries

1. You will be using the same colorimeter system you used in Experiment 2. You will need to use the **green** LED . . . this is critical!

2. A CdS cell, described in Lessons 3, 4, and 5 of your Using Computers book from Chem 135, is the light detector. You should send somewhere between 1500 and 2000 millivolts to the DAC in order to measure a reasonable transmission current, I. You must also "SEND On TO LED-A" in order to make the green LED light up when properly connected.

3. You will need to design an experiment program which will send transmission current measurements and time data to a **Spreadsheet** file. The program must run until the transmission current no longer significantly changes. This will probably be about 90 seconds. You will want to take data at least every one second.

4. You must design the experiment program so it will not take data until the formic acid has been added to the bromine water.

Procedures

1. Using a graduated pipette, add 3.00 mL of Br_2(aq) to a clean, dry plastic cuvette.

2. With the cuvette in the colorimeter, *carefully* add 1 drop of 6 M HCOOH to the cuvette, cover the colorimeter, and start your program. As time progresses, the Br_2 will react with the HCOOH and the color of the Br_2 will fade.

3. Once the experiment is complete, use the **Spreadsheet** to convert the transmission currents into [Br_2].

4. You must *at least* obtain a plot of [Br_2] versus time.

5. You will probably want to repeat the experiment at least two more times.

6. Following the discussion on pp 525-529 from Chang, determine as much information as possible about the kinetics of the bromine/formic acid reaction *using your data*. This includes reaction rates and rate constant information, if possible.

Minimum Requirements

1. You must design an experiment which will send photocell readings and time data to a data file while the bromine/formic acid reaction is occurring.

2. You must use the **Spreadsheet** to *at least* obtain a plot of $[Br_2]$ versus time.

3. You must present as much information as possible about the kinetics of the bromine/formic acid reaction.

4. You will submit one report (by you and your lab partner). Your lab instructor will inform you of the exact time and date the lab report is due.

References

Chang, R. Chemistry, 3rd ed. New York: Random House, Inc., 1988.

CHEM 136
FINAL PROJECT #2
THERMOMETRIC DETERMINATION OF CHLORIDE
CONCENTRATION IN WATER
Difficulty Factor: 1.05

Introduction/Objective

You will determine the concentration of chloride in two unknown solutions by thermometric titration with silver nitrate ($AgNO_3$).

Background

Chloride ion (Cl^-) is one of the major inorganic ions found in water and wastewater. In drinking water, the salty taste produced by chloride concentrations is variable and depends on the chemical contents of the water. Some water samples containing 250 mg chloride/liter may have a detectable salty taste if the cation is sodium. However, if the cations are calcium and magnesium, the salty taste may be absent in concentrations up to 1000 mg chloride/liter. High chloride content in water can be detrimental to metallic pipes and structural material, as well as plants.

Standard Methods details several methods for determining Cl^- concentrations in water. Several involve determining a color endpoint through titration. However, these methods require considerable solution preparation and considerable practice. The methods are quite accurate (better than 10 mg/L) in the 100-1000 mg chloride/L range.

If the chloride ion concentration is higher than 1000 mg/L, a much easier and faster method of determining chloride ion concentration is by a *thermometric titration*. Quite simply, the endpoint of the titration is determined by examining the changing slope of the temperature versus volume of titrant curve. For this titration, the chloride-containing sample is titrated with a known concentration of silver nitrate ($AgNO_3$). The following reaction occurs is the unknown chloride solution has the sodium cation:

(1) $AgNO_3(aq) + NaCl(aq) \longrightarrow AgCl(s) + NaNO_3$

As the above reaction proceeds, a white precipitate forms. In addition, the reaction is exothermic, so heat is evolved. As heat is evolved, the temperature of the reaction mixture will rise. When the reaction is complete, heat is no longer evolved and the temperature of the mixture will stabilize. The point at which the temperature stabilizes is the endpoint. It is important to note that the temperature rise associated with the amounts of solution you will use is quite small, but ideally suited for measurement by a thermistor and the lab interface.

Experimental

Lab Interface Preliminaries

1. You will be using a thermistor to measure temperature. You should calibrate the thermistor using ice water and hot tap water.

2. You will need to design an experiment program which will send temperature and time (or volume) measurements to a **Spreadsheet** data file. You may also wish to actively graph the information on the screen while the experiment is running.

Procedures

1. Using a 500-mL volumetric flask, prepare a 0.05 M NaCl solution. Calculate the concentration of your solution to at least three significant figures. Pour about half of the solution into a clean, dry beaker and pour the remainder of the solution down the drain. This will be your "practice" solution.

2. Titrate 20.0 mL (measured with a pipette) of your "practice" NaCl solution with 0.05 M $AgNO_3$. *Note the exact concentration of the silver nitrate on the bottle!* The titration set-up is shown in Figure 1. You have two options in performing the titration, the first option being the better:

   a. Perform a timed titration, as discussed in paragraph 4b, page 53 of your Chem 136 lab book. Remember, you are measuring temperature and not pH. When the experiment is complete, you can use the **Spreadsheet** to convert time into volume, using a measured flow rate. This technique takes a little practice! It is important to continue stirring the solution throughout the titration.

   b. Perform a manual titration, as discussed in paragraph 4a on page 53. The disadvantage of a manual titration is that it takes longer between measurements, increasing the likelihood of temperature instabilities.

3. Once you're convinced you can see the thermometric endpoint at the "expected" point for your practice solution, proceed to titrate two of the available unknown solutions. You must repeat each titration at least twice and take an average endpoint to determine the unknown chloride concentrations.

FIGURE 1. Set-up for thermometric titration.

Minimum Requirements

1. You must design an experiment which will send temperature and time (or volume) to a data file during the titration.

2. You must use the **Spreadsheet** to obtain temperature versus volume plots for each titration. From the plots, you will determine the endpoint and subsequently the chloride concentration of the unknowns.

3. Calculate the chloride ion concentration for each unknown in units of *molarity* and *mg chloride/liter of solution*.

4. Using heat of formation data (e.g. from a textbook or CRC handbook), determine the heat of reaction for the reaction of $AgNO_3$ and $NaCl$. Based on this value for ΔH_{rxn} and the actual moles of reactants used in each of your titrations, calculate the *theoretical* amount of heat evolved in each titration.

5. You will submit one report (by you and your lab partner). Your lab instructor will inform you of the exact time and date the lab report is due.

References

Chang, R. Chemistry, 3rd ed. New York: Random House, Inc., 1988.

Standard Methods for the Examination of Water and Wastewater, 15th ed.,
    Washington D.C.: American Public Health Association, 1980.

CHEM 136
FINAL PROJECT #3
DETERMINATION OF AMMONIA IN HOUSEHOLD CLEANER
Difficulty Factor: 1.05

Introduction/Objective

You will be using thermometric and potentiometric acid/base titration techniques to determine the percentage of ammonia in two common liquid household cleaners.

Background

Before you begin this project, it is very important that you have a very good understanding of acids, bases, pH, and titrations. A review of Experiments 3 and 4 in your Chem 136 lab book would be helpful!

Acids and bases are chemical compounds which we encounter constantly in our everyday lives. It is the acid/base properties of many household products which cause them to work. For example, in Parsons Clear Ammonia household cleaner, the major active ingredient is *aqueous ammonia*. Ammonia acts as a surfactant and makes non-polar substances, such as grease, at least partially soluble in water. Aqueous ammonia is often the ingredient of choice in products that claim to be "grease cutters". Aqueous ammonia is actually a mixture of $NH_3(aq)$ and water. The resulting solution is basic because the NH_3 acts as a base, accepting protons from water:

(1) $NH_3(aq) + H_2O(l) \longrightarrow NH_4^+(aq) + OH^-(aq)$ $K_b = 1.8 \times 10^{-5}$

To determine the mass percent of ammonia in a household cleaner, you can titrate a known mass of the cleaner with a strong acid, such as HCl:

(2) $NH_3(aq) + HCl(aq) \longrightarrow NH_4^+(aq) + Cl^-(aq)$
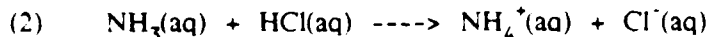
The pH at the equivalence point will be less than 7 for such a titration because of hydrolysis:
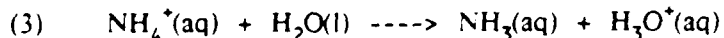
(3) $NH_4^+(aq) + H_2O(l) \longrightarrow NH_3(aq) + H_3O^+(aq)$

The equivalence point can be determined potentiometrically, as you did in Experiment #4 for acetic acid. However, the equivalence point may also be found *thermometrically*. Reaction (2) is exothermic, so heat is evolved during the titration. As heat is evolved, the temperature of the reaction mixture will rise. When the equivalence point is reached, reaction (2) is complete and heat is no longer evolved. The temperature of the mixture will stabilize, rising at a

significantly slower rate or possibly dropping a bit. The point at which the
temperature curve stabilizes or significantly changes slope is the equivalence point.
It is important to note that the temperature rise associated with the amounts of
solution you will use is relatively small, but ideally suited for measurement by a
thermistor and the lab interface.

In this project, you will quantitatively determine the percentage of
ammonia in two popular liquid household cleaners. You will determine the
equivalence points both potentiometrically and thermometrically.

Experimental

Lab Interface Preliminaries

1. You will be using a thermistor to measure temperature. You should
calibrate the thermistor using ice water and tap water. You must also calibrate the
electrode using pH 7 buffer before using.

2. You will need to design an experiment program which will send
temperature, pH, and volume (or time) measurements to a **Spreadsheet** data file.
You may also wish to actively graph the pH versus volume (or time) information
on the screen while the experiment is running.

Procedures

1. To obtain good temperature data, you will perform the titration in two
nested styrofoam cups, as shown in Figure 1. You will be using 0.1 M HCl as the
titrant. *Note the exact concentration of HCl on the bottle!*

2. **You** must determine how large of a sample (from a mass standpoint) of
the household cleaner to use in order to get a "good" equivalence point. This will
involving some thinking or "trial and error" on your part. *Do not use straight
cleaner! You must dilute however much you use by at least a factor of 5 with
distilled water!* Your nested styrofoam cup must contain at least 30 mL of diluted
cleaner to ensure that the pH electrode is fully submerged.

3. Titrate the above solution with 0.1 M HCl. *Note the exact concentration
of the HCl on the bottle!* The titration set-up is shown in Figure 1. You have two
options in performing the titration:

a. Perform a timed titration, as discussed in paragraph 4b, page 53 of your Chem 136 lab book. Remember, you are measuring pH *and* temperature in this project. When the experiment is complete, you can use the Spreadsheet to convert time into volume, using a measured flow rate. This technique may take a little practice! It is important to continue stirring the solution throughout the titration.

b. Perform a manual titration, as discussed in paragraph 4a on page 53. The disadvantage of a manual titration is that it takes longer between measurements, increasing the likelihood of temperature instabilities. However, if the manual titration is done carefully and reasonably quickly, you will get good results. The solution must be stirred before making each measurement.

4. You should titrate a sample of each household cleaner *at least* twice, averaging the thermometric results and averaging the potentiometric results to obtain a value for the mass percent of ammonia in each cleaner.
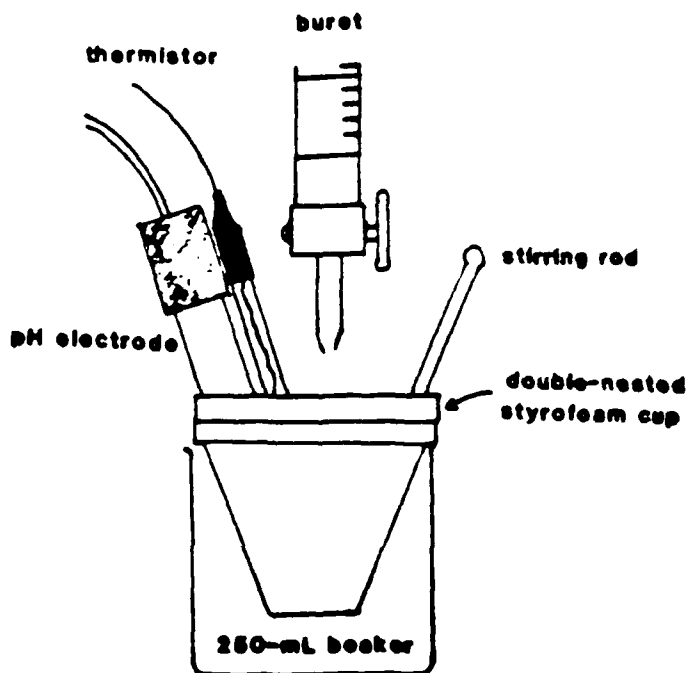
FIGURE 1. Set-up for titration of household cleaner.

Minimum Requirements

1. You must design an experiment which will send pH readings, temperature and total volumes of HCl added (or, times) to a data file for the liquid household cleaner titrations. You will hand in a listing of the program lines which perform the task.

2. You must use the **Spreadsheet** to obtain plots to determine the equivalence points for your titrations. At a minimum, you must include the following plots for each titration:

        a. pH versus volume of HCl and the derivative of the pH vs volume curve plotted on the same graph.

        b. pH and temperature versus volume plotted on the same graph.

        c. temperature versus volume of HCl and the derivative of the pH vs volume curve plotted on the same graph.

3. Include a discussion and/or data and calculations which show the size of household ammonia sample you determined would work best for your titrations.

4. Calculate the mass percent of ammonia in both household cleaners using the potentiometric *and* thermometric equivalence points. Compare these results. *You should make more than one titration for each household cleaner and average your final results for mass percent for each household cleaner.*

5. Using the heat of formation data (e.g. from a textbook or CRC handbook), determine the heat of reaction for the reaction of $NH_3(aq)$ and $HCl(aq)$. Based on this value for ΔH_{rxn} and the actual moles of reactants used in each of your titrations, calculate the *theoretical* amount of heat evolved in each titration.

6. You will submit <u>one</u> report (by you and your lab partner). Your lab instructor will inform you of the exact time and date the lab report is due.

· CHEM 136
FINAL PROJECT #4
FORMATION OF COMPLEX IONS OF CADMIUM
Difficulty Factor: 1.02

Introduction/Objective

You will determine the formula of the coordination complex solution specie formed between cadmium (Cd^{2+}) and iodide ion (I^-).

Background

Many of the transition metals can form *complex ions* in solution. For example, zinc can form a number of complex ions with many different species:

$$Zn^{2+}(aq) + 4NH_3(aq) \longrightarrow Zn(NH_3)_4^{2+}(aq)$$

$$Zn^{2+}(aq) + Cl^-(aq) \longrightarrow ZnCl^+(aq)$$

$$Zn^{2+}(aq) + 4CN^-(aq) \longrightarrow Zn(CN)_4^{2-}(aq)$$
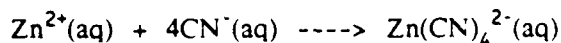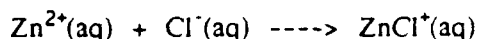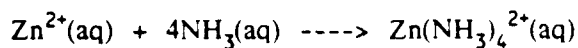
In fact, many transition metals can form more than one relatively stable complex ion with the same anion. If we let M^{2+} represent the metal cation, the following reactions could occur with the anion I^-:

$$M^{2+}(aq) + I^-(aq) \longrightarrow MI^+(aq)$$

$$MI^+(aq) + I^-(aq) \longrightarrow MI_2(aq)$$

$$MI_2(aq) + I^-(aq) \longrightarrow MI_3^-(aq)$$

$$MI_3^-(aq) + I^-(aq) \longrightarrow MI_4^{2-}(aq)$$

$$MI_4^{2-}(aq) + I^-(aq) \longrightarrow MI_5^{3-}(aq), \text{ and so on.}$$
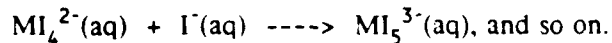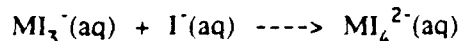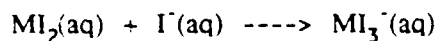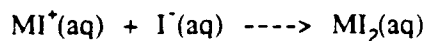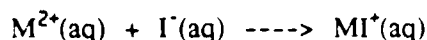
But how can one determine which complex ions are actually formed? Spectroscopic techniques, such as Raman spectroscopy, can identify the ions that are present in solution. Another much simpler, but less versatile, technique for identifying these complex ions is through *thermometric analysis*.

The previous complex ions have associated with them a *heat of formation.* If the formation of the products are thermodynamically favorable, heat is released, causing a temperature rise in the solution. As long as sufficient I^- is provided, heat will be released and stable products will form. This process will continue until stable products are no longer formed, resulting in a ceasing of the temperature rise.

In theory, the complex ion formation reactions will have different heats of reaction. Therefore, it could be possible to monitor the sequential formation of the stable complex ions as more iodine is added by examining the varying slope of a 'temperature' versus 'volume of iodine added' curve. In reality, such an effect is not visible due to the changing volume of the solution and the small difference in heats of reaction. However, it *is* possible to observe when the reaction ceases to release heat from a temperature leveling or drop. It is at this point that a formula for a thermodynamically stable complex ion can be determined based on the amount of available metal cation and the amount of added anion. In this project, you will determine the formula for a stable complex ion formed from the reaction of cadmium ion with iodide, or
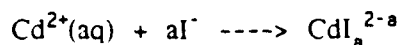
$$Cd^{2+}(aq) + aI^- \longrightarrow CdI_a^{2-a}$$

Experimental

Lab Interface Preliminaries

1. You will be using a thermistor to measure temperature. You should calibrate the thermistor using ice water and hot tap water.

2. You will need to design an experiment program which will send temperature and time (or volume) measurements to a **Spreadsheet** data file. You may also wish to actively graph the information on the screen while the experiment is running.

Procedures

Safety Note: *Cadmium compounds are toxic if inhaled or ingested. Place all waste solutions containing cadmium into the appropriately marked container. At the end of the lab period. make sure you wash your hands!*

1. Using a pipette, place a 10.0 mL sample of 1.0 M Cd^{2+} solution (*note the exact concentration on the bottle*) into a double-nested styrofoam cup and place the cup assembly into a 250-mL beaker, as shown in Figure 1. Place a calibrated thermistor into the solution.

2. Titrate the 10.0 mL sample of Cd^{2+} with 2.0 M NaI. *Note the exact concentration of the sodium iodide on the bottle!* You will be monitoring the temperature of the solution during the _ration. The titration set-up is shown in Figure 1. You have two options in performing the titration, the first option being the better.

   a. Perform a timed titration, as discussed in paragraph 4b, page 53 of your Chem 136 lab book. Remember, you are measuring temperature and not pH. When the experiment is complete, you can use the **Spreadsheet** to convert time into volume, using a measured flow rate. This technique takes a little practice! It is important to continue stirring the solution throughout the titration.

   b. Perform a manual titration, as discussed in paragraph 4a on page 53. The disadvantage of a manual titration is that it takes longer between measurements, increasing the likelihood of temperature instabilities.

3. Repeat the titration *at least* once. Average the results to determine the formula for the cadmium-iodide complex ion.

4. Repeat the entire experiment using 1.0 M NaI. You should prepare your 1.0 M NaI from the 2.0 M stock solution (you should need no more than 200 mL of 1.0 M NaI). Compare your results with those using the 2.0 M NaI.
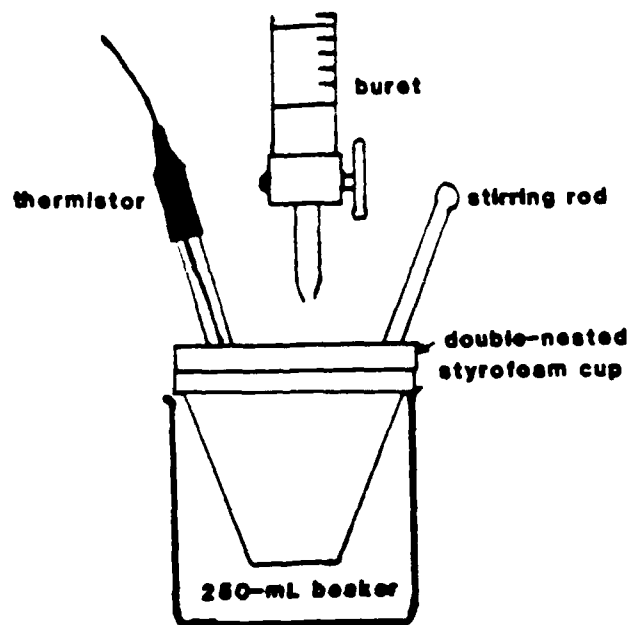
FIGURE 1. Set-up for thermometric titration.

Minimum Requirements

1. You must design an experiment which will send temperature and time (or volume) to a data file during the titration.

2. You must use the **Spreadsheet** to obtain temperature versus volume plots for each titration. From the plots, you will determine the endpoint and subsequently the formula for the stable cadmium-iodide complex.

3. Based on your data, write the balanced reaction for the formation of the stable cadmium-iodide complex.

4. You will submit one report (by you and your lab partner). Your lab instructor will inform you of the exact time and date the lab report is due.

References

Chang, R. Chemistry, 3rd ed. New York: Random House, Inc., 1988.

Hill, J.O. and Magee, R.J. "Advanced Undergraduate Experiments in Thermoanalytical Chemistry," J. Chem. Educ., **1988**, 65 (11), 1024-1026.

Cotton, F.A. and Wilkinson, G. Advanced Inorganic Chemistry, 4th ed. New York: John Wiley & Sons, Inc., 1980.

CHEM 136
FINAL PROJECT #5
USING A THERMOMETRIC INDICATOR TO DETERMINE
ACETATE ION CONCENTRATIONS IN UNKNOWNS
Difficulty Factor: 1.02

Introduction/Objective

You will determine the concentration of acetate ions in unknown solutions by using the thermometric indicator SO_4^{2-}.

Background

Thermometric titrimetry is a very useful analytical technique to determine endpoints in a titration. In a conventional titration, a rapid pH rise (or fall) or a color indicator is used to detect the endpoint. In a thermometric titration, the heat of reaction is the indicator. Many titrations may not give a detectable endpoint with a color indicator and thus are ideally suited for a thermometric titration.

One ion which is suitable for detection through thermometric titrimetry is the acetate ion (CH_3COO^-), which will be abbreviated as Ac^-. When acetate ion, a weak base, is titrated with a strong acid such as HCl, the following neutralization reaction occurs:

$$H_3O^+(aq) + Ac^-(aq) ----> HAc(aq) + H_2O(l)$$
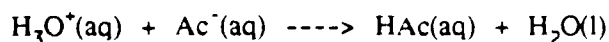
The above reaction has zero enthalpy; the ΔH_{rxn} is equal to zero. Therefore, as the neutralization of the Ac^- proceeds, no heat is released; the temperature of the reaction mixture stays constant. If sulfate ion (SO_4^{2-}) is also present in the solution, the H_3O^+ will preferentially react with the Ac^- first, then react with the SO_4^{2-} as follows:

$$H_3O^+(aq) + SO_4^{2-}(aq) ----> HSO_4^-(aq) + H_2O(l)$$
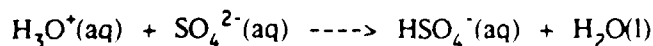
This reaction is endothermic, with a ΔH_{rxn} of +19.7 kJ/mole. At the end of the acetate neutralization (the endpoint), the above reaction will begin to occur, resulting in a drop in temperature of the reaction mixture. The SO_4^{2-} is acting as a thermometric indicator, causing a different temperature-volume profile at the acetate endpoint. Using sulfate as a thermometric indicator and HCl as the titrant, you will determine the concentration of acetate ion in unknown solutions. You will compare these results with the potentiometric endpoints.

Experimental

Lab Interface Preliminaries

1. You will be using a thermistor to measure temperature. You should calibrate the thermistor using ice water and hot tap water. You must also calibrate the pH electrode with pH 7 buffer before using.

2. You will need to design an experiment program which will send temperature, pH, and volume (or time) measurements to a **Spreadsheet** data file. You may also wish to actively graph the pH or temperature information on the screen while the experiment is running.

Procedures

1. To obtain good temperature data, you will perform the titration in two nested styrofoam cups, as shown in Figure 1. You will be using 0.3 M HCl as the titrant. *Note the exact concentration of HCl on the bottle!*
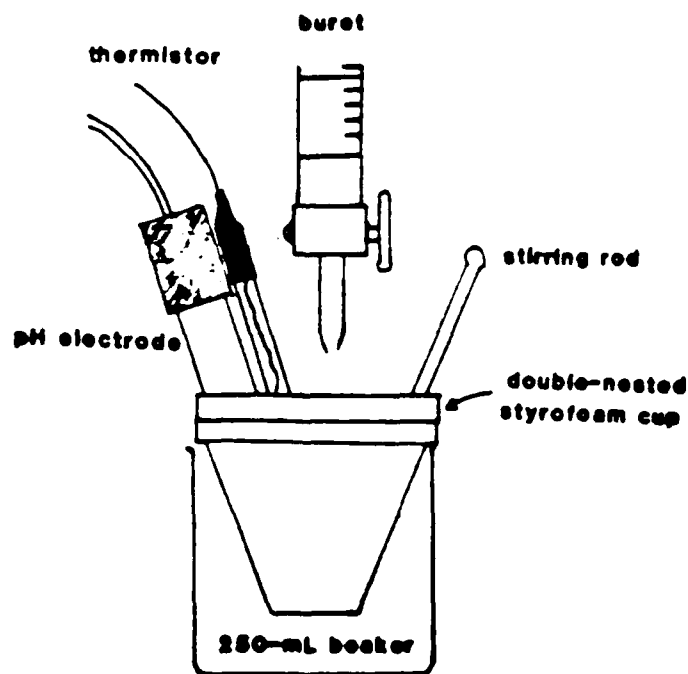
FIGURE 1. Set-up for determination of acetate ion concentration.

2. Using a pipette, place 20.0 mL of one of the unknowns (you will do two unknowns) into the double-nested styrofoam cup. Then, using a graduated cylinder, add 20 mL of 0.2 M Na_2SO_4 to the unknown in the cup.

3. Titrate the unknown sample of acetate ion with 0.3 M HCl. *Note the exact concentration of the HCl on the bottle!* You will be monitoring the temperature of the solution *and* the pH during the titration. The titration set-up is shown in Figure 1. You have two options in performing the titration:

   a. Perform a timed titration, as discussed in paragraph 4b, page 53 of your Chem 136 lab book. Remember, you are measuring temperature and pH. When the experiment is complete, you can use the **Spreadsheet** to convert time into volume, using a measured flow rate. This technique takes a little practice! It is important to continue stirring the solution throughout the titration.

   b. Perform a manual titration, as discussed in paragraph 4a on page 53. The disadvantage of a manual titration is that it takes longer between measurements, increasing the likelihood of temperature instabilities.

4. You should titrate each of the two unknowns you choose *at least* twice. You should then average the thermometric results and compare these with the average of the potentiometric (pH curve) results for each unknown.

5. **Optional:** There will be a bottle of sodium acetate available so that you can make 100-mL of your own, "known" acetate ion solution. By titrating 20.0 mL of your "known" solution, you can "anticipate" an endpoint during a practice titration. This will give you practice with respect to your titration technique and what to look for during the titration of your two unknowns.

Minimum Requirements

1. You must design an experiment which will send temperature, pH and time (or volume) to a data file during the titration. You will hand in a listing of the program lines which perform the task.

2. You must use the **Spreadsheet** to obtain plots to determine the equivalence points for your titrations. At a minimum, you must include the following plots for each titration:

   a. pH versus volume of HCl and the derivative
      of the pH vs volume curve plotted on
      the same graph.

   b. pH and temperature versus volume plotted on
      the same graph.

c. Temperature versus volume of HCl and the
derivative of the pH versus volume curve
plotted on the same graph.

3. Using the thermometric endpoints, determine the concentration (M) of
acetate ions in two of the unknown solutions. In addition, determine the
concentration of acetate ion concentration in the unknowns using the
potentiometric equivalence point, if possible.

4. You will submit one report (by you and your lab partner). Your lab
instructor will inform you of the exact time and date the lab report is due.

References

Chang, R. Chemistry, 3rd ed. New York: Random House, Inc., 1988.

Hill, J.O. and Magee, R.J. "Advanced Undergraduate Experiments in
Thermoanalytical Chemistry," J. Chem. Educ., 1988, 65 (11), 1024-1026.

CHEM 136
FINAL PROJECT #6
PHOSPHORIC ACID CONCENTRATIONS IN COLAS
Difficulty Factor: 1.00

Introduction/Objective

You will determine the phosphoric acid concentration in two cola products using acid/base titration techniques.

Background

Before you begin this project, it is very important that you have a very good understanding of acids, bases, pH, and titrations. A review of Experiments 3 and 4 in your Chem 136 lab book would be helpful!

Acids and bases are chemical compounds which we encounter constantly in our everyday lives. It is the acid content of many foods and beverages which contribute significantly to the taste. Soft drinks often contain varying quantities of several acids; in cola products, these acids are predominantly carbonic acid (from the carbonated water) and phosphoric acid. By decarbonating the cola, you can determine the phosphoric acid concentration through titrimetric analysis. The phosphoric acid content is usually quite low, so good technique is critical.

Experimental

Lab Interface Preliminaries

1. You will be using a pH electrode to measure pH. You must calibrate the electrode with pH 7 buffer before using.

2. You will need to design an experiment program which will send pH and volume (or time) measurements to a **Spreadsheet** data file. You may also wish to actively graph the pH versus volume (or time) information on the screen while the experiment is running.

3. You will also need to design a simple program that will measure the pH of a solution, independent of time or volume of titrant. You will use this simple program to measure the pH of the cola before and after decarbonation.

Procedures

1. Pour about 100 mL of cola into a clean, dry 250-mL beaker. Measure the pH of the cola at room temperature. To prepare the cola for titration, it must first be decarbonated. Cover the beaker containing the cola with a watch glass and boil for about 20 minutes. This will remove the carbon dioxide from the cola, which would interfere with the phosphoric acid titration. Measure the pH of the decarbonated cola after it has cooled to room temperature.

2. Using a 25-mL volumetric pipette, place 25.0 mL of room temperature, decarbonated cola into a 150-mL t ker. Place a *calibrated* pH electrode and a glass stirring rod into the beaker with the cola.

3. Titrate the cola with 0.01 M NaOH. *Note the exact concentration of the NaOH on the bottle!* You have two options in performing the titration:

a. Perform a timed titration, as discussed in paragraph 4b, page 53 of your Chem 136 lab book. When the experiment is complete, you can use the **Spreadsheet** to convert time into volume, using a measured flow rate. This technique may take a little practice! It is important to continue stirring the solution throughout the titration.

b. Perform a manual titration, as discussed in paragraph 4a on page 53. The only disadvantage of a manual titration is that you must be careful to make small volume increments around the endpoints. However, if the manual titration is done carefully, you will get good results. The solution must be stirred before making each measurement.

4. You should be able to see two equivalence points, one for each of the following reactions:

$$H_3PO_4(aq) + OH^-(aq) \longrightarrow H_2PO_4^-(aq) + H_2O(l)$$

$$H_2PO_4^-(aq) + OH^-(aq) \longrightarrow HPO_4^{2-}(aq) + H_2O(l)$$
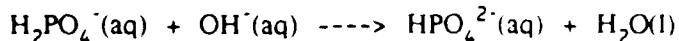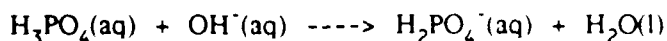
5. Using the data from the first equivalence point, calculate the concentration of phosphoric acid in the cola. You must titrate each of two colas *at least* twice, averaging the results.

6. Using all of your titration curves, determine K_{a1} and K_{a2} for phosphoric acid, averaging the resu*** from all of your titration curves. *Note: Experiment 4 in your Chem 136 la· ·ok explains how to do this for weak acids!*

Minimum Requirements

1. You must design an experiment which will send pH readings and total volumes of NaOH added (or, times) to a data file for the cola titrations. You will hand in a listing of the program lines which perform the task. **Since this program will likely be similar to ones you have previously design, you must ensure that the program is very user-friendly.** You must also design a simple experiment which will allow you to measure the pH of the cola before and after decarbonation. *You do not have to hand in a copy of the simple program.*

2. You must use the **Spreadsheet** to obtain plots to determine the equivalence points for your titrations. At a minimum, you must include a plot of pH versus volume of NaOH and the derivative of the pH vs volume curve plotted on the same graph. You must include these plots for each titration.

3. Using the first equivalence point, calculate the concentration of phosphoric acid in each of the two colas you chose.

4. From your graphs, determine the first and second acid dissociation constants (K_{a1} and K_{a2}) for phosphoric acid.

5. You will submit one report (by you and your lab partner). Your lab instructor will inform you of the exact time and date the lab report is due.

References

Amend, J.R. Chemistry. Montana State University, 1988.

Chang, R. Chemistry, 3rd ed. New York: Random House, Inc., 1988.

Murphy, J. "Determination of Phosphoric Acid in Cola Beverages." J.Chem.Educ., **1983,** 60 (5), 420-421.

CHEM 136
FINAL PROJECT #7
DETERMINATION OF ACID IN TOILET BOWL CLEANERS
Difficulty Factor: 1.00

Note: *You cannot do this project if you did the project titled "Acid / Base Properties of Common Household Products and Food" in Chem 135! Please choose another project if this is the case!*

Introduction/Objective

You will be using thermometric and potentiometric acid/base titration techniques to determine the percentage of hydrochloric acid in two common liquid toilet bowl cleaners.

Background

Before you begin this project, it is very important that you have a very good understanding of acids, bases, pH, and titrations. A review of Experiments 3 and 4 in your Chem 136 lab book would be helpful!

Acids and bases are chemical compounds which we encounter constantly in our everyday lives. It is the acid/base properties of many household products which cause them to work. For example, in Lysol and Vanish liquid toilet bowl cleaners, the major active ingredient is HCl. The significant reaction occurring in the toilet bowl is the following:

$$2HCl(aq) + CaCO_3(s) \longrightarrow 2Cl^-(aq) + Ca^{2+}(aq) + H_2O(l) + CO_2(g)$$
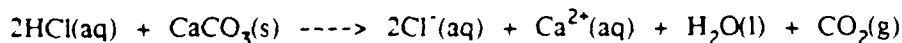
The $CaCO_3(s)$ is commonly known as toilet bowl "scale". The HCl, through an acid/base reaction, literally dissolves the scale. The HCl also serves as a disinfectant, killing many bacteria and germs due to the drastic pH change once the cleaner is in the toilet bowl. Other chemical bacteria killers are also often added to toilet bowl cleaners. Still other ingredients are used to enhance the flow properties.

To determine the mass percent of HCl in the toilet bowl cleaner, you can titrate a known mass of the bowl cleaner with sodium hydroxide, NaOH:
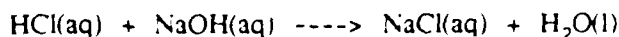
$$HCl(aq) + NaOH(aq) \longrightarrow NaCl(aq) + H_2O(l)$$

The equivalence point can be determined potentiometrically, as you did in Experiment #4 for acetic acid. However, the equivalence point may also be found *thermometrically*. The above reaction is exothermic, so heat is evolved. As heat is evolved, the temperature of the reaction mixture will rise. When the reaction is

complete, heat is no longer evolved and the temperature of the mixture will stabilize and perhaps even begin to fall. The point at which the temperature curve stabilizes or significantly changes slope is also the equivalence point. It is important to note that the temperature rise associated with the amounts of solution you will use is relatively small, but ideally suited for measurement by a thermistor and the lab interface.

In this project, you will quantitatively determine the percentage of hydrochloric acid in two popular liquid toilet bowl cleaners. You will determine the equivalence point both potentiometrically and thermometrically.

Experimental

Lab Interface Preliminaries

1. You will be using a thermistor to measure temperature. You should calibrate the thermistor using ice water and tap water. You must also calibrate the electrode using pH 7 buffer before using.

2. You will need to design an experiment program which will send temperature, pH, and volume (or time) measurements to a **Spreadsheet** data file. You may also wish to actively graph the pH versus volume (or time) information on the screen while the experiment is running.

Procedures

1. To prepare the bowl cleaner for titration, add 5.0 mL of liquid bowl cleaner (using a buret or pipette) to a dry, clean, previously double-styrofoam cup. Weigh the cups and bowl cleaner to obtain the mass of the bowl cleaner. After you've determined the mass of the bowl cleaner, add about 40 mL of distilled water.

2. Titrate the above solution with 0.5 M NaOH. *Note the exact concentration of the NaOH on the bottle!* The titration set-up is shown in Figure 1. You have two options in performing the titration:

a. Perform a timed titration, as discussed in paragraph 4b, page 53 of your Chem 136 lab book. Remember, you are measuring pH *and* temperature in this project. When the experiment is complete, you can use the **Spreadsheet** to convert time into volume, using a measured flow rate. This technique may take a little practice! It is important to continue stirring the solution throughout the titration.

b. Perform a manual titration, as discussed in paragraph 4a on page 53. The disadvantage of a manual titration is that it takes longer between measurements, increasing the likelihood of temperature instabilities. However, if the manual titration is done carefully and reasonably quickly, you will get good results. The solution must be stirred before making each measurement.

3. You should titrate a sample of each toilet bowl cleaner *at least* twice, averaging the thermometric results and averaging the potentiometric results to obtain a value for the mass percent of HCl in each toilet bowl cleaner.
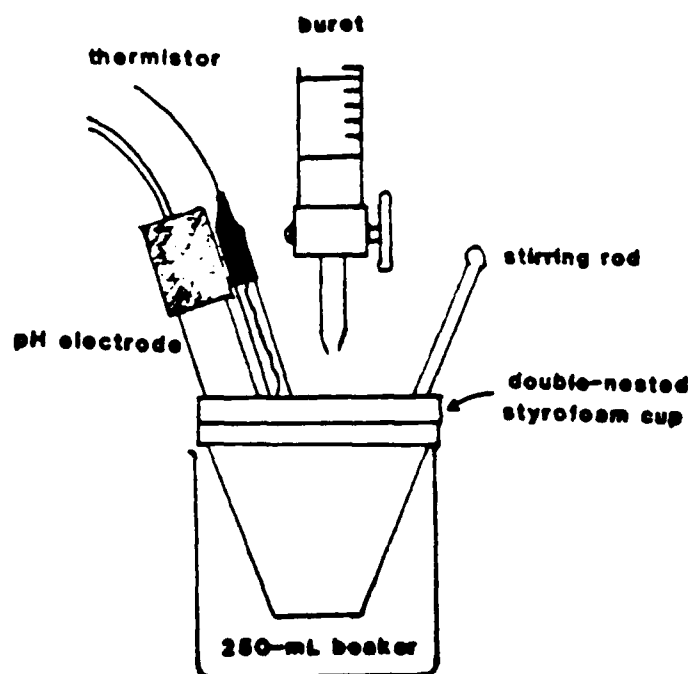
FIGURE 1. Set-up for titration of toilet bowl cleaner.

Minimum Requirements

1. You must design an experiment which will send pH readings, temperature and total voiumes of NaOH added (or, times) to a data file for the liquid bowl cleaner titrations. You will hand in a listing of the program lines which perform the task.

2. You must use the **Spreadsheet** to obtain plots to determine the equivalence points for your titrations. At a minimum, you must include the following plots for each titration:

    a. pH versus volume of NaOH and the derivative of the pH vs
        volume curve plotted on the same graph.

    b. pH and temperature versus volume plotted on the same graph.

    c. Temperature versus volume of NaOH and the derivative of the
        pH vs volume curve plotted on the same graph.

    3 Calculate the mass percent of hydrochloric acid in both bowl cleaners
using the potentiometric *and* thermometric equivalence points. Compare these
results. *You should make more than one titration for each bowl cleaner and average
your final results for mass percent for each bowl cleaner.*

    4. Using the heat of formation data (e.g. from a textbook or CRC
handbook), determine the heat of reaction for the reaction of HCl and NaOH.
Based on this value for ΔH_{rxn} and the actual moles of reactants used in each of
your titrations, calculate the *theoretical* amount of heat evolved in each titration.

    5. You will submit <u>one</u> report (by you and your lab partner). Your lab
instructor will inform you of the exact time and date the lab report is due.

References

Amend, J.R. Chemistry. Montana State University, 1988.

FINAL REPORT REQUIREMENTS

Do not write your final reports in a blue book. Write them on regular notebook paper or type them. **Typing is preferred,** but not mandatory. You and your lab partner will submit one report. Your lab instructor will inform you of the exact due date and time.

The following sections MUST be included in your report. Each section of your report should begin with the following headings. This requirement is to establish consistency among reports for grading purposes. Grammar, spelling, and neatness do count!

A. Purpose

B. Experimental Measurements and Data

1. Include a description/drawing of your experimental set-up.
2. Present your data in a format that displays the data clearly. The exact format is your call!
3. Include a printout of your computer program in this section of your report.

C. Data Analysis/Calculations

Any calculations using your data must be shown in this section. This will vary considerably from project to project.

D. Discussion of Results

This is a very important section, but not necessarily lengthy. You should discuss how you got your results and what the results mean. You should also discuss any problems you may have had.

E. Conclusions

Summarize your results, both quantitative and qualitative. This will likely be simply a restatement of information from the previous sections.

F. References

Document any outside references you used in preparing your report. You may use any book, journal, or person as a reference. The people in your lab section and your lab instructor do not have to be documented as references.

APPENDIX C

EXPERIMENT PROGRAMS FOR THE MSU LAB INTERFACE

EXPERIMENT PROGRAMS FOR THE MSU LAB INTERFACE

The following programs were developed to support a series of workshops held from January 1990 to April 1990 for Montana teachers. Workshop participants were given a short write-up on each project (Reference 87) very similar to the format in Appendices A and B. They then designed programs similar to those that follow to accomplish the objectives of the projects. Programs for the following projects are included in this appendix:

Earth Science

> #1: Heat Capacity of Materials
>
> #2: Comparison of Insulation Materials
>
> #3: Sediment Concentration in Water

Biology

> #4: Hamster Activity
>
> #5: Human Reaction Rates for Unanticipated and Anticipated Events
>
> #6: Growth Curve of a Yeast Colony

Physics

> #7: Ohm's Law
>
> #8: Determination of Planck's Constant
>
> #9: Falling Body Experiment

Chemistry

> #10: E'ectrochemistry
>
> #11: Potentiometric and Thermometric Titration of Toilet Bowl Cleaner
>
> #12: Determination of Beta Radiation Penetration

\#1. _Heat Capacity of Materials_. There is considerable latitude in designing an experiment to determine the heat capacity of materials. The following experiment program simply measures the temperature of a material every 30 seconds for up to one hour and stores the data in a **Spreadsheet** file.

```
 1 CLEAR TEXT SCREEN
 2 PRINT "Project 1:  Heat Capacity of Materials" ON LINE 1
 3 PRINT "This program measures the temperature of" ON LINE 5
 4 PRINT "a substance with respect to time." ON LINE 6
 5 PRINT "Time and temperature data will be sent" ON LINE 8
 6 PRINT "to a data file every 30 seconds" ON LINE 9
 7 PRINT "for up to one hour." ON LINE 10
 8 PRINT "Press the "w-button" to start taking data." ON LINE 20
 9 WAIT UNTIL INPUT FROM SwW = Pressed
10 CLEAR TEXT SCREEN
11 START Timer 1 FOR sec
12 PRINT "Time (sec)" ON LINE 7
13 PRINT "Temperature" ON LINE 15
14 PRINT "Flip up the "x-switch" to stop before one hour." ON LINE 20
15 START Repeat Timer FOR sec
16 SEND INPUT FROM Timer1 TO COL-A
17 SEND INPUT FROM Temp TO COL-B
18 PRINT INPUT FROM Timer1 ON LINE 8
19 PRINT INPUT FROM Temp ON LINE 16
20 IF INPUT FROM SwX = On GOTO 22
21 REPEAT FROM LINE 16 EVERY 30 sec FOR 120 TIMES
22 CLEAR TEXT SCREEN
23 PRINT "End of experiment." ON LINE 12
24 STOP
```

FIGURE 85. Experiment Program to Determine Heat Capacity of Materials.

#2. **Comparison of Insulation Materials.** This program is identical to the one
in Figure 85. However, there are *many* ways to design and actually perform this
experiment. This is a classic example of an experiment that is conceptually easy to
understand but lends itself to being accomplished in a wide variety of methods due to
the many variables involved. The actual experimental set-up is crucial for obtaining
reproducible and comparable data.

```
1 CLEAR TEXT SCREEN
2 PRINT "Project 2:  Comparison of Insulation Materials" ON LINE 1
3 PRINT "This program measures the temperature under" ON LINE 5
4 PRINT "insulation material with respect to time." ON LINE 6
5 PRINT "Time and temperature data will be sent" ON LINE 8
6 PRINT "to a data file every 30 seconds" ON LINE 9
7 PRINT "for up to one hour." ON LINE 10
8 PRINT "Press the "w-button" to start taking data." ON LINE 20
9 WAIT UNTIL INPUT FROM SwW = Pressed
10 CLEAR TEXT SCREEN
11 START Timer1 FOR sec
12 PRINT "Time (sec)" ON LINE 7
13 PRINT "Temperature" ON LINE 15
14 PRINT "Flip up the "x-switch" to stop before one hour." ON LINE 20
15 START Repeat Timer FOR sec
16 SEND INPUT FROM Timer1 TO COL-A
17 SEND INPUT FROM Temp TO COL-B
18 PRINT INPUT FROM Timer1 ON LINE 8
19 PRINT INPUT FROM Temp ON LINE 16
20 IF INPUT FROM SwX = On GOTO 22
21 REPEAT FROM LINE 16 EVERY 30 sec FOR 120 TIMES
22 CLEAR TEXT SCREEN
23 PRINT "End of experiment." ON LINE 12
24 STOP
```

FIGURE 86. Experiment Program for the Comparison of Insulation Materials.

#3. Sediment Concentration in Water. This is an outstanding experiment for the MSU lab interface because there are so many ways to design an effective experiment. The program in Figure 87 is designed to measure relative or absolute concentrations of sediment in water samples using a LED as a light source and a CdS photocell as a light detector. In reality, a monochromatic light source is unnecessary and a light bulb is probably an even better light source. A styrofoam block and a large test tube make an excellent "colorimeter" for this experiment.

```
1 CLEAR WHOLE SCREEN
2 PRINT "Project 3a:  Relative Sediment Concentration" ON LINE 1
3 PRINT "This program measures light to a CdS cell." ON LINE 3
4 PRINT "The photocell must be connected to DAC and 1." ON LINE 6
5 PRINT "The LED must be connected to the " ON LINE 7
6 PRINT "green and white posts marked "A"." ON LINE 8
7 PAUSE
8 SEND On TO LED-A
9 SEND 1000.000000 TO DAC
10 CLEAR TEXT SCREEN
11 PRINT "Input a sample # or conc. when prompted." ON LINE 20
12 PRINT "This value will go to Col-A of a data file." ON LINE 21
13 SEND INPUT FROM Kbd TO COL-A
14 CLEAR TEXT SCREEN
15 PRINT "When you press <return>, you'll see a current" ON LINE 4
16 PRINT "indicating the amount of light through the sample." ON LINE 5
17 PRINT "Press the "F" key on the keyboard to send" ON LINE 10
18 PRINT "the value on the screen to Col-B of your" ON LINE 11
19 PRINT "Spreadsheet data file." ON LINE 12
20 PAUSE
21 PRINT "CURRENT" LARGE ON LINE 1
22 PRINT "Press the "F" key to take data." ON LINE 20
23 PRINT INPUT FROM I LARGE ON LINE 2
24 IF INPUT FROM LastKey = 102.000000 GOTO 26
25 GOTO 23
26 SEND INPUT FROM I TO COL-B
27 CLEAR TEXT SCREEN
28 PRINT "Do you want data on another sample?" ON LINE 1
29 PRINT "Enter a '1' for yes, '2' for no." ON LINE 2
30 IF INPUT FROM Kbd = 1.000000 GOTO 10
31 SEND Off TO LED-A
32 CLEAR TEXT SCREEN
33 PRINT "End of experiment." ON LINE 12
34 STOP
```

FIGURE 87. Experiment Program to Measure Sediment Concentration in Water.

235

The program in Figure 88 is designed to measure sedimentation rates in a sample; that is, how the transmission current of a photocell varies with time. This is very similar to the program described in Chapter 6 which measures the transmission currents during the bromine/formic acid reaction.

```
 1 CLEAR TEXT SCREEN
 2 PRINT "Project 3b: Sedimentation Rates" ON LINE 1
 3 PRINT "This program measures light transmission to" ON LINE 3
 4 PRINT "a photocell with respect to time." ON LINE 4
 5 PRINT "The photocell must be connected to DAC and I." ON LINE 6
 6 PRINT "The LED must be connected to the" ON LINE 7
 7 PRINT "green and white posts marked "A"." ON LINE 8
 8 PRINT "When you hit <return>, the program will begin" ON LINE 19
 9 PRINT "taking current & time data every 3 sec for 10 min." ON LINE 20
10 PAUSE
11 SEND On TO LED-A
12 SEND 1000.000000 TO DAC
13 CLEAR TEXT SCREEN
14 START Timer1 FOR sec
15 PRINT "Flip up the "x-switch" to stop experiment early." ON LINE 20
16 PRINT "Time (sec)" ON LINE 6
17 PRINT "Transmission current" ON LINE 12
18 START Repeat Timer FOR sec
19 SEND INPUT FROM Timer1 TO COL-A
20 SEND INPUT FROM I TO COL-B
21 PRINT INPUT FROM Timer1 ON LINE 7
22 PRIN' INPUT FROM I ON LINE 13
23 IF INPUT FROM SwX = On GOTO 25
24 REPEAT FROM LINE 19 EVERY 3 sec FOR 200 TIMES
25 CLEAR TEXT SCREEN
26 PRINT "End of experiment." ON LINE 12
27 STOP
```

FIGURE 88. Experiment Program to Measure Sedimentation Rates.

#4. Hamster Activity. The activity of a hamster can be easily measured since they like to spend much of their waking time running on a treadmill. We designed an experiment in which the treadmill rotated through a photogate, which measured the number of revolutions of the wheel during any time period. This activity can also be monitored with respect to the temperature of the room and the amount of light in the room. This experiment is conceptually easy to visualize, but requires a great deal of thought to actually do it; an ideal project for the MSU lab interface!

```
1 CLEAR TEXT SCREEN
2 PRINT "Project 4:  Hamster Activity" ON LINE 1
3 PRINT "This program measures the activity of" ON LINE 3
4 PRINT "a hamster on a treadmill as a function of" ON LINE 4
5 PRINT "light in the room." ON LINE 5
6 PRINT "Attach the photogate to LED-A and Photo-1." ON LINE 7
7 PRINT "Attach the photocell to DAC and I." ON LINE 9
8 PRINT "The program will send a time to Col-A every" ON LINE 12
9 PRINT "time the photogate goes "dark"." ON LINE 13
10 PRINT "A photocell current will then be sent to Col-B." ON LINE 15
11 PRINT "Press <return> to begin taking data." ON LINE 20
12 PAUSE
13 CLEAR TEXT SCREEN
14 SEND On TO LED-A
15 SEND 500.000000 TO DAC
16 START Timer1 FOR sec
17 PRINT "Time (sec)" ON LINE 6
18 PRINT "Photocell current" ON LINE 12
19 PRINT "Flip up the "x-switch" to stop." ON LINE 20
20 IF INPUT FROM SwX = On GOTO 27
21 WAIT UNTIL INPUT FROM PD1 = Dark
22 SEND INPUT FROM Timer1 TO COL-A
23 SEND INPUT FROM I TO COL-B
24 PRINT INPUT FROM COL-A ON LINE 7
25 PRINT INPUT FROM COL-B ON LINE 13
26 GOTO 20
27 CLEAR TEXT SCREEN
28 PRINT "Your experiment has stopped." ON LINE 12
29 STOP
```

FIGURE 89. Experiment Program to Measure Hamster Activity.

#5. <u>Human Reaction Rates for Unanticipated and Anticipated Events</u>. This

project probably requires the most ingenuity of any of the twelve projects in this

Appendix. The designer must truly understand what it is they want to do before they

attempt this project. For the first part of the project, they must design an experiment

which measures the reaction time of a person for an unanticipated event. In the

program in Figure 90, the "event" is the appearance of the word **GO** on the screen in a

"random" fashion, requesting the subject to respond by pushing the w-button on the

interface box. The same 8-second delay (Line 18) is sufficient time to prevent the

waiting subject from anticipating the event.

```
1 CLEAR TEXT SCREEN
2 PRINT "Project 5a: Reaction Rate for Unanticipated Event" ON LINE 1
3 PRINT "This program measures the time it takes" ON LINE 5
4 PRINT "for a person to respond to an unanticipated" ON LINE 6
5 PRINT "visual stimulus." ON LINE 7
6 PRINT "You will be asked to enter a Trial Number." ON LINE 12
7 PRINT "After typing the Trial # and pressing <return>," ON LINE 13
8 PRINT "wait until you see the word "GO" on the screen." ON LINE 14
9 PRINT "When you see the word "GO" appear, press the" ON LINE 15
10 PRINT ""w-button" on the interface box." ON LINE 16
11 PRINT "The Trial Number and Reaction Time will be sent" ON LINE 18
12 PRINT "to Col-A and Col-B, respectively, of a data file." ON LINE 19
13 PAUSE
14 CLEAR TEXT SCREEN
15 PRINT "Enter the trial number." ON LINE 20
16 SEND INPUT FROM Kbd TO COL-A
17 CLEAR TEXT SCREEN
18 DELAY 8 sec
19 PRINT "GO" ON LINE 12
20 START Timer1 FOR sec
21 WAIT UNTIL INPUT FROM SwW = Pressed
22 SEND INPUT FROM Timer1 TO COL-B
23 PRINT "Reaction Time (Seconds)" ON LINE 16
24 PRINT INPUT FROM COL-B ON LINE 17
25 PRINT "Do you want to try again?" ON LINE 19
26 PRINT "Enter a '1' for yes, '2' for no." ON LINE 20
27 IF INPUT FROM Kbd = 1.000000 GOTO 14
28 CLEAR TEXT SCREEN
29 PRINT "End of experiment." ON LINE 12
30 STOP

26
```

FIGURE 90. Program to Measure Reaction Time for an Unanticipated Event.

In Figure 91, the program is designed to measure the reaction time of a subject anticipating an event. The anticipated event is 5 asterisks (* * * * *) appearing on the screen. Successive asterisks appear on the screen at one second intervals, so the appearance of the 5 asterisks can be anticipated. When actually tested, the reaction time in the experiment in Figure 90 is longer than the reaction time for the experiment in Figure 91.

```
 1 CLEAR TEXT SCREEN
 2 PRINT "Project 5b: Reaction Rate for Anticipated Event" ON LINE 1
 3 PRINT "This program measures the time it takes" ON LINE 5
 4 PRINT "for a person to respond to an anticipated" ON LINE 6
 5 PRINT "visual stimulus." ON LINE 7
 6 PRINT "YOU will be asked to enter a Trial Number." ON LINE 12
 7 PRINT "After typing in the Trial # and pressing <return>," ON LINE 13
 8 PRINT "wait until 5 asterisks (* * * * *) appear." ON LINE 14
 9 PRINT "When you see * * * * * appear, press the" ON LINE 15
10 PRINT "'w-button" on the interface box." ON LINE 16
11 PRINT "The Trial Number and Reaction Time will be sent" ON LINE 18
12 PRINT "to Col-A and Col-B, respectively, of a data file." ON LINE 19
13 PAUSE
14 CLEAR TEXT SCREEN
15 PRINT "Enter the trial number." ON LINE 20
16 SEND INPUT FROM Kbd TO COL-A
17 CLEAR TEXT SCREEN
18 DELAY 2 sec
19 PRINT "*" ON LINE 1
20 DELAY 1 sec
21 PRINT "* *" ON LINE 4
22 DELAY 1 sec
23 PRINT "* * *" ON LINE 7
24 DELAY 1 sec
25 PRINT "* * * *" ON LINE 10
26 DELAY 1 sec
27 PRINT "* * * * *" ON LINE 13
28 START Timer1 FOR SEC
29 WAIT UNTIL INPUT FROM SwW = Pressed
30 SEND INPUT FROM Timer1 TO COL-B
31 PRINT "Reaction Time (Seconds)" ON LINE 16
32 PRINT INPUT FROM COL-B ON LINE 17
33 PRINT "Do you want to try again?" ON LINE 19
34 PRINT "Enter a '1' for yes, '2' for no." ON LINE 20
35 IF INPUT FROM Kbd = 1.000000 GOTO 14
36 CLEAR TEXT SCREEN
37 PRINT "End of experiment." ON LINE 12
38 STOP
```

FIGURE 91. Program to Measure Reaction Time for an Anticipated Event.

#6. Growth Curve of a Yeast Colony. The experimental set-up for this project is very similar to that for the sedimentation concentration in water. A large test tube containing the yeast colony in a sugar solution is placed in a container which allows light (a LED or light bulb) to shine through the test tube to a detector (CdS cell). Since the yeast produce carbon dioxide, it is useful to monitor the pH of the solution at the same time.

```
1 CLEAR WHOLE SCREEN
2 PRINT "Project 6:  Growth Curve of a Yeast Colony" ON LINE 1
3 PRINT "This program measure light transmission to a" ON LINE 3
4 PRINT "photocell and pH with respect to time." ON LINE 4
5 PRINT "The photocell must be connected to DAC and I." ON LINE 6
6 PRINT "The LED must be connected to the" ON LINE 7
7 PRINT "green and white posts marked "A"." ON LINE 8
8 PRINT "When you hit the <return> key, the program will" ON LINE 19
9 PRINT "begin taking current, pH, & time data every " ON LINE 20
10 PRINT "10 minutes for 24 hours unless you flip up "x"." ON LINE 21
11 PAUSE
12 SEND 1000.000000 TO DAC
13 CLEAR TEXT SCREEN
14 START Timer1 FOR MIN
15 PRINT "Flip up the "x-switch" to stop experiment early." ON LINE 21
16 PRINT "It may take up to 10 min. a response." ON LINE 22
17 PRINT "Time (minutes)" ON LINE 6
18 PRINT "Transmission current" ON LINE 12
19 PRINT "pH" ON LINE 18
20 START Repeat Timer FOR min
21 SEND INPUT FROM Timer1 TO COL-A
22 SEND INPUT FROM I TO COL-B
23 SEND INPUT FROM pH TO COL-C
24 PRINT INPUT FROM Timer1 ON LINE 7
25 PRINT INPUT FROM I ON LINE 13
26 PRINT INPUT FROM pH ON LINE 19
27 IF INPUT FROM SwX = On GOTO 29
28 REPEAT FROM LINE 21 EVERY 10 min FOR 144 TIMES
29 CLEAR TEXT SCREEN
30 PRINT "End of experiment." ON LINE 12
31 STOP
```

FIGURE 92. Program to Measure the Growth Curve of a Yeast Colony.

#7. Ohm's Law. In the Ohm's Law project, the experimenter determines the resistance of a series of unknown resistors by obtaining a series of current measurement with different voltages applied across the resistor. Using Ohm's Law ($V = IR$), the resistance can be calculated from the slope of a voltage/current plot.

```
 1 CLEAR TEXT SCREEN
 2 PRINT "Project 7:  Ohm's Law" ON LINE 1
 3 PRINT "This program increments voltages across a resistor" ON LINE 5
 4 PRINT "and sends the voltage and current to a data file." ON LINE 6
 5 PRINT "Voltages (mV) are sent to Col-A." ON LINE 8
 6 PRINT "Currents (microamps) are sent to Col-B." ON LINE 9
 7 PRINT "Voltage will be incremented by 100 mV every 5 s" ON LINE 11
 8 PRINT "and, along with current, displayed on the screen." ON LINE 12
 9 PRINT "The program stops when the voltage >= 2000 mV." ON LINE 13
10 PRINT "Connect your resistor to DAC and I at this time." ON LINE 20
11 PAUSE
12 CLEAR TEXT SCREEN
13 PRINT "Voltage (mV)" ON LINE 1
14 PRINT "Current (microamps)" ON LINE 5
15 INCREMENT DAC BY 100.000000 MILLIVOLTS
16 DELAY 5 sec
17 SEND INPUT FROM DAC TO COL-A
18 SEND INPUT FROM I TO COL-B
19 PRINT INPUT FROM COL-A ON LINE 2
20 PRINT INPUT FROM COL-B ON LINE 6
21 IF INPUT FROM DAC >= 2000.000000 GOTO 23
22 GOTO 15
23 CLEAR TEXT SCREEN
24 PRINT "End of experiment." ON LINE 12
25 STOP
```

FIGURE 93. Experiment Program for Finding an Unknown Resistance.

#8. Determination of Planck's Constant. In this experiment, Planck's constant is determined by measuring the voltage at which current first flows through a LED of known emission wavelength. Using the equation $E = h\nu$, Planck's constant h can then be calculated. The experiment makes use of the *reverse photoelectric effect*, seeing when light is first emitted rather than when electrons are first emitt￼ ·o determine Planck's constant. In Figure 94, the voltage to the DAC is stepped up until the current through the LED exceeds a "noise threshold" current of 0.1 microamps.

```
 1 CLEAR TEXT SCREEN
 2 PRINT "Project 8:  Determination of Planck's Constant," ON LINE 1
 3 PRINT "current-determined cutoff voltage for a LED." ON LINE 2
 4 PRINT "This program increments the voltage applied" ON LINE 5
 5 PRINT "across a LED connected to DAC an I by 20 mV" ON LINE 6
 6 PRINT "until a current greater than 0.1 microamps flows," ON LINE 7
 7 PRINT "indicating the onset of emission from the LED." ON LINE 8
 8 PRINT "Connect the LED before continuing." ON LINE 20
 9 PAUSE
10 CLEAR TEXT SCREEN
11 PRINT "Voltage (mV)" ON LINE 1
12 PRINT "Current (microamps)" ON LINE 5
13 INCREMENT DAC BY 20.000000 MILLIVOLTS
14 PRINT INPUT FROM DAC ON LINE 2
15 PRINT INPUT FROM I ON LINE 6
16 IF INPUT FROM I >= 0.100000 GOTO 18
17 GOTO 13
18 PRINT "Write down the cutoff voltage." ON LINE 17
19 PRINT "Do you want to try again?" ON LINE 19
20 PRINT "Enter a '1' for yes, '2' for no." ON LINE 20
21 IF INPUT FROM Kbd = 1.000000 GOTO 10
22 CLEAR TEXT SCREEN
23 PRINT "End of experiment." ON LINE 12
24 STOP
```

FIGURE 94. Experiment Program for Determining Planck's Constant.

#9. The Falling Body Experiment. The falling body experiment is also a very thought-intensive experiment to design. The photogates must react very quickly, since the entire experiment normally takes less than one second. Proper choice of fast commands is essential. In this version of the experiment, two photogates are used and the time it takes for a falling object to move from one photogate to the other is timed. The photogates must be properly aligned before starting the experiment, ensuring the receiver "sees" the transmitter. Figure 95 is a simple program to check the alignment of the photogates.

```
1 CLEAR TEXT SCREEN
2 PRINT "Project 9a: Falling Body Experiment" ON LINE 1
3 PRINT "This simple program tests for photogate alignment." ON LINE 2
4 PRINT "Move the photogates until the word "light" appears" ON LINE 5
5 PRINT "on the screen." ON LINE 6
6 PRINT "Attach Photogate 1 to Photo-1 and LED-A and/or" ON LINE 8
7 PRINT "attach Photogate 2 to Photo-2 and LED-B." ON LINE 9
8 PAUSE
9 SEND On TO LED-A
10 SEND On TO LED-B
11 CLEAR TEXT SCREEN
12 PRINT "Photogate 1" ON LINE 9
13 PRINT "Photogate 2" ON LINE 15
14 PRINT "Press the "w-button" to stop the test." ON LINE 22
15 PRINT INPUT FROM PD1 ON LINE 10
16 PRINT INPUT FROM PD2 ON LINE 16
17 IF INPUT FROM SwW = Released GOTO 15
18 SEND Off TO LED-A
19 SEND Off TO LED-B
20 CLEAR TEXT SCREEN
21 PRINT "End of test." ON LINE 12
22 STOP
```

FIGURE 95. Program to Check Photogate Alignment.

The program in Figure 96 can be used to measure the acceleration due to gravity for a body initially at rest at Photogate 1, and falling a known distance to Photogate 2.

```
1 CLEAR TEXT SCREEN
2 PRINT "Project 9b:  Falling Body Experiment" ON LINE 1
3 PRINT "This program uses two photogates to determine" ON LINE 2
4 PRINT "the acceleration due to gravity using an object" ON LINE 3
5 PRINT "initially at rest, blocking Photogate 1 (PD1)." ON LINE 6
6 PRINT "When PD1 becomes "light", a timer will start." ON LINE 8
7 PRINT "When the object passes through PD2, which is" ON LINE 9
8 PRINT "lined up directly below PD1 at a distance h," ON LINE 10
9 PRINT "PD2 will go "dark" and stop the timer, sending" ON LINE 11
10 PRINT "the time to Col-B." ON LINE 12
11 PAUSE
12 SEND On TO LED-A
13 SEND On TO LED-B
14 CLEAR TEXT SCREEN
15 PRINT "Enter the distance between photogates (h)." ON LINE 20
16 SEND INPUT FROM Kbd TO COL-A
17 CLEAR TEXT SCREEN
18 PRINT "With the object blocking PD1, drop the object." ON LINE 1
19 WAIT UNTIL INPUT FROM PD1 = Light
20 START Timer1 FOR sec
21 WAIT UNTIL INPUT FROM PD2 = Dark
22 SEND INPUT FROM Timer1 TO COL-B
23 CLEAR TEXT SCREEN
24 PRINT "time of fall (seconds)" ON LINE 10
25 PRINT INPUT FROM Timer1 ON LINE 11
26 PRINT "Do you wish to change the distance, h," ON LINE 18
27 PRINT "and try again?" ON LINE 19
28 PRINT "Enter a '1' for yes, '2' for no."ON LINE 20
29 IF INPUT FROM Kbd = 1.000000 GOTO 14
30 SEND Off TO LED-A
31 SEND OFF TO LED-B
32 CLEAR TEXT SCREEN
33 PRINT "End of experiment." ON LINE 12
34 STOP
```

FIGURE 96. Program to Determine the Acceleration Due to Gravity.

#10. Electrochemistry. As part of the electrochemistry project, the experimenter must design a chemical battery which produces at least 1.5 volts while lighting a red LED for at least 5 minutes. The data must be stored at approximately one second intervals to verify that the battery meets operational specifications. Figure 97 shows the experiment program needed to accomplish the task.

```
1 CLEAR TEXT SCREEN
2 PRINT "Project 10:  Electrochemistry" ON LINE 1
3 PRINT "This program displays time (s) and voltage (mV)" ON LINE 5
4 PRINT "data and sends time to Col-A and voltage (E) to" ON LINE 6
5 PRINT "Col-B of a data file." ON LINE 7
6 PRINT "Data will be sent every one second until 5 minutes" ON LINE 9
7 PRINT "have expired or until the x-switch is flipped up." ON LINE 10
8 PRINT "which ever comes first." ON LINE 11
9 PRINT "The program will begin when you hit <return>." ON LINE 17
10 PAUSE
11 CLEAR TEXT SCREEN
12 START Timer1 FOR sec
13 PRINT "Flip up the x-switch to stop before 5 min." ON LINE 20
14 PRINT INPUT FROM Timer1 LARGE ON LINE 1
15 PRINT INPUT FROM E LARGE ON LINE 2
16 SEND INPUT FROM Timer1 TO COL-A
17 SEND INPUT FROM E TO COL-B
18 IF INPUT FROM SwX = On GOTO 22
19 IF INPUT FROM Timer1 >= 300.000000 GOTO 22
20 DELAY 1 sec
21 GOTO 14
22 CLEAR TEXT SCREEN
23 PRINT "End of experiment." ON LINE 12
24 STOP
```

FIGURE 97. Program to Measure the Voltage of a Designed Battery.

#11. Potentiometric and Thermometric Titration of Toilet Bowl Cleaner. This
program is virtually identical to the titration program described by Figure 40 in
Chapter 6. The program given in Figure 98 is for a timed titration; a manual titration,
as described in Figure 41, will work as well.

```
1 CLEAR TEXT SCREEN
2 PRINT "Project 11:  pH and Temperature Titration" ON LINE 1
3 PRINT "of Toilet Bowl Cleaner, timed titration." ON LINE 2
4 PAUSE
5 PRINT "This program is used to measure pH & Temp" ON LINE 1
6 PRINT "versus time for an acid/base titration." ON LINE 2
7 PRINT "Before starting the experiment, obtain a" ON LINE 4
8 PRINT "constant flow rate with your buret into" ON LINE 5
9 PRINT "a separate beaker, at 2 to 5 drops/sec." ON LINE 6
10 PRINT "Once you've established a flow rate," ON LINE 9
11 PRINT "push the flask which contains your unknown" ON LINE 10
12 PRINT "acid under the buret." ON LINE 11
13 PRINT "PRESS THE "W" BUTTON on the interface" ON LINE 14
14 PRINT "to start the experiment once the" ON LINE 15
15 PRINT "titrant hits the unknown!" ON LINE 16
16 PRINT "Flip up the "x" switch to stop" ON LINE 20
17 PRINT "the experiment." ON LINE 21
18 WAIT UNTIL INPUT FROM SwW = Pressed
19 CLEAR TEXT SCREEN
20 PRINT "pH" LARGE ON LINE 1
21 PRINT "Temperature" ON LINE 18
22 PRINT "Flip up the "x" switch to stop." ON LINE 21
23 START Timer FOR sec
24 PRINT INPUT FROM pH LARGE ON LINE 2
25 PRINT INPUT FROM Temp ON LINE 19
26 SEND INPUT FROM Timer1 TO COL-A
27 SEND INPUT FROM pH TO COL-B
28 SEND INPUT FROM Temp TO COL-C
29 IF INPUT FROM SwX = On GOTO 32
30 DELAY 250 millisec
31 GOTO 24
32 CLEAR TEXT SCREEN
33 PRINT "End of experiment." ON LINE 12
34 STOP
```

FIGURE 98. Program for the Timed Titration of Toilet Bowl Cleaner.

246

#12. Determination of Beta Radiation Penetration. This experiment is the

same as discussed in Chapter 6 and shown as Figure 56. The experiment program is

shown here as Figure 99 for completeness.

```
1 CLEAR TEXT SCREEN
2 PRINT "Project 12:  Beta Radiation Penetration" ON LINE 1
3 PAUSE
4 PRINT "In this experiment, you will attempt to" ON LINE 1
5 PRINT "find out how much material must be used" ON LINE 2
6 PRINT "to absorb or stop the beta radiation" ON LINE 3
7 PRINT "from a radioactive sample of uranium." ON LINE 4
8 PRINT "You will collect data for this portion" ON LINE 6
9 PRINT "of the experiment by placing between" ON LINE 7
10 PRINT "0 and 60 index cards between the source" ON LINE 8
11 PRINT "and the Geiger tube." ON LINE 9
12 PRINT "The program will ask you to input the" ON LINE 11
13 PRINT "number of cards between the Geiger tube" ON LINE 12
14 PRINT "and the source." ON LINE 13
15 PRINT "Once you've entered that value." ON LINE 15
16 PRINT "the program will count for 30 sec." ON LINE 16
17 PRINT "The number of cards will be entered in" ON LINE 18
18 PRINT "Column A of your Spreadsheet file." ON LINE 19
19 PRINT "The number of counts will be entered in" ON LINE 20
20 PRINT "Column B of your Spreadsheet file." ON LINE 21
21 PAUSE
22 CLEAR TEXT SCREEN
23 PRINT "Enter the total number of cards." ON LINE 20
24 SEND INPUT FROM Kbd TO COL-A
25 CLEAR TEXT SCREEN
26 RESET COUNTER
27 START Timer1 FOR sec
28 PRINT INPUT FROM Counter LARGE ON LINE 1
29 PRINT INPUT FROM Timer1 LARGE ON LINE 2
30 IF INPUT FROM Timer1 >= 30.000000 GOTO 32
31 GOTO 28
32 SEND INPUT FROM Counter TO COL-B
33 SEND INPUT FROM COL-A TO GraphX
34 SEND INPUT FROM COL-B TO GraphY
35 PRINT "Are you finished taking data?" ON LINE 20
36 PRINT "Enter a '1' for yes, a '2' for no." ON LINE 21
37 IF INPUT FROM Kbd = 2.000000 GOTO 22
38 CLEAR TEXT SCREEN
39 PRINT "End of experiment." ON LINE 12
40 STOP
```

FIGURE 99. Program for Determining Beta Radiation Penetration.